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**U.S. Army  
Environmental  
Center**

**TEST PLAN  
for  
DEVELOPMENT AND DEMONSTRATION  
of  
HOT GAS DECONTAMINATION  
FOR EXPLOSIVES**

**at  
HAWTHORNE ARMY AMMUNITION PLANT  
Hawthorne, Nevada 89415-0015**

***Prepared for*  
U.S. ARMY ENVIRONMENTAL CENTER  
Aberdeen Proving Ground, Maryland 21010-5401**

**DTIC QUALITY INSPECTED**

***Prepared by*  
THE TENNESSEE VALLEY AUTHORITY  
ENVIRONMENTAL RESEARCH CENTER  
Muscle Shoals, Alabama 35660-1010**

**May 1994**

**TVA Contract No. N87179V  
Report No. SFIM-AEC-TS-CR-94027**

**19961016 006**

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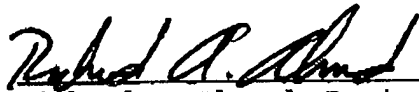
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This document was prepared by the Tennessee Valley Authority Environmental Research Center, under TVA contract TV-87179V, and approved by

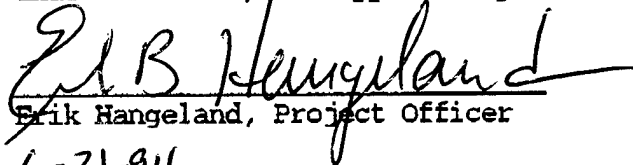


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6-20-94

Date

for the U. S. Army Environmental Center, Aberdeen Proving Grounds, Maryland, under MIPR4753, and approved by



Erik Hangeland, Project Officer

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Date



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## REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS		
SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION / AVAILABILITY OF REPORT Unlimited		
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			5. MONITORING ORGANIZATION REPORT NUMBER(S) SFIM-AEC-TS-CR-94027		
6a. NAME OF PERFORMING ORGANIZATION Tennessee Valley Authority	6b. OFFICE SYMBOL (If applicable) CEB 4C	7a. NAME OF MONITORING ORGANIZATION U.S. Army Environmental Center			
6c. ADDRESS (City, State, and ZIP Code) TVA Reservation Post Office Box 1010 Muscle Shoals, Alabama 35660-1010		7b. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Grounds, Maryland 21010-5401			
8a. NAME OF FUNDING / SPONSORING ORGANIZATION U.S. Army Environmental Center	8b. OFFICE SYMBOL (If applicable) SFIM-AEC-TSD	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER TVA Contract No. TV-87179			
8c. ADDRESS (City, State, and ZIP Code) Aberdeen Proving Grounds, Maryland 21010-5401		10. SOURCE OF FUNDING NUMBERS			
		PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Test Plan for Development and Demonstration of Hot Gas Decontamination for Explosives at Hawthorne Army Ammunition Plant					
12. PERSONAL AUTHOR(S) J. R. Watson, M. C. Crim, C. W. Brown, J. J. Hoagland, W. J. Rogers					
13a. TYPE OF REPORT Final	13b. TIME COVERED FROM _____ TO _____	14. DATE OF REPORT (Year, Month, Day) May 1994		15. PAGE COUNT	
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP			
			Hot Gas Decontamination		
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified		
22a. NAME OF RESPONSIBLE INDIVIDUAL Erik Hangeland		22b. TELEPHONE (Include Area Code) (301) 671-2054		22c. OFFICE SYMBOL SFIM-AEC-TSD	

## ABBREVIATIONS

106mm	- U.S. Army, 106 millimeter artillery projectile
175mm	- U.S. Army, 175 millimeter artillery projectile
3"/5"	- U.S. Navy Projectile
ALSS	- Analytical Laboratory Support Services (TVA, Resource Group at Muscle Shoals, Alabama)
CEM	- Continuous Emissions Monitor
CO	- Carbon Monoxide
CO <sub>2</sub>	- Carbon Dioxide
Comp B	- Explosive Made From TNT, RDX, and Wax
Comp A-3	- Explosive Made From RDX and Wax
CRREL	- Cold Regions Research and Engineering Laboratory
DRE	- Destruction and Removal Efficiency
DZB	- Day and Zimmerman/Basil Corporation, operating contractor at HWAAP
EPA	- Environmental Protection Agency
FF-13	- Flash Furnace Facility at HWAAP
GLP-0001	- TVA/ALSS Laboratory Procedure Preparation
GLP-0003	- TVA/ALSS Procedure Preparation and Distribution
GLP-0005	- TVA/ALSS Nonconformances and Corrective Actions
GLP-0007	- TVA/ALSS Control of Changes to Software
H&S	- Health and Safety
HBX	- Explosive Made From TNT, RDX, Aluminum, Lecithin, and Wax
HGD	- Hot Gas Decontamination
HWAAP	- Hawthorne Army Ammunition Plant, Hawthorne, Nevada
ID	- Induced Draft
lb	- pounds
MDL	- Method Detectable Limits
MK 9	- U.S. Navy Depth Charge
MK 25	- U.S. Navy Ship Mine
MK 54	- U.S. Navy Depth Bomb
MM5	- Modified Method 5 - EPA Sampling Procedure
MSDS	- Material Safety Data Sheet
MX	- Matrix Spike or Matrix Spike Duplicate Recovery Was Outside Limits Due to Suspected Matrix Effects
NA	- Compound Not Analyzed
NAD	- Naval Ammunition Depot

NO <sub>x</sub>	- Nitrogen Oxides
OBOD	- Open Burning-Open Detonation
ppm	- Parts Per Million
QA/QC	- Quality Assurance/Quality Control
RCRA	- Resource Conservation and Recovery Act
RDX	- Hexahydro - 1, 3, 5 - trinitro - 1, 3, 5 - triazine
RFW	- Roy F. Weston
SARM	- Standard Analytical Reference Material
SD	- Surrogate Recovery Low Due to Dilution
SO <sub>x</sub>	- Sulfur Oxides
SM	- Surrogate Recovery Out of Limits, Matrix Effect Suspected
SP-001	- TVA/ALSS Sample Chain of Custody Procedure
TNT	- 2, 4, 6 - Trinitrotoluene
TR	- Compound Present At Trace Level
TVA	- The Tennessee Valley Authority
USADACS	- U.S. Army Defense Ammunition Center and School
USAEC	- U.S. Army Environmental Center
USAEHA	- U.S. Army Environmental and Health Agency
WADF	- Western Area Demilitarization Facility
Yellow D	- Ammonium Picrate
°C	- Degrees Celsius
°F	- Degrees Fahrenheit

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## **1.0 INTRODUCTION**

### **1.1 Background**

Figure 1 is a map of the west-central portion of the state of Nevada showing the location of Hawthorne. Hawthorne Army Ammunition Plant (HWAAP) covers some 246,000 acres of land situated in Mineral County approximately 135 miles southeast of Reno, Nevada, along U.S. Route 95. HWAAP was originally constructed in 1929 as a U.S. Navy Ammunition Depot (NAD). The Western Area Demilitarization Facility (WADF) was constructed by the Navy from 1975 to 1977 to dispose of Naval munitions in a controlled manner (Reference #1). The U.S. Army continued the Navy's munitions demilitarization program and assumed control of Hawthorne in October 1977 and changed the name to Hawthorne Army Ammunition Plant.

The WADF, on the northern end of the installation boundary near Walker Lake, is shown in Figure 2.

The WADF conducts a process of reverse assembly of the munitions followed by steam, autoclave, mechanical, or washout removal of explosive charges. Reclaimed explosives can, at least theoretically, be recycled, or employed in less demanding applications such as demolitions or as donor explosives for safe disposal of unexploded ordinance. Small metal parts are decontaminated by furnace flashing. Large items could be decontaminated in Building 117-15. This flashing chamber was originally designed and constructed to provide for flashing large items. The parts are then released as scrap metal. A detailed view of the WADF is shown in Figure 3.

An enclosed flashing chamber was constructed at WADF for the purpose of decontaminating items by flashing them with surplus propellant powder. This approach was not successful and the chamber has been adapted to the Hot Gas Decontamination Process (HGD) which has been demonstrated to decontaminate individual items under test conditions. Figure 4 shows the area of the flashing furnace and other support buildings used for this test plan.



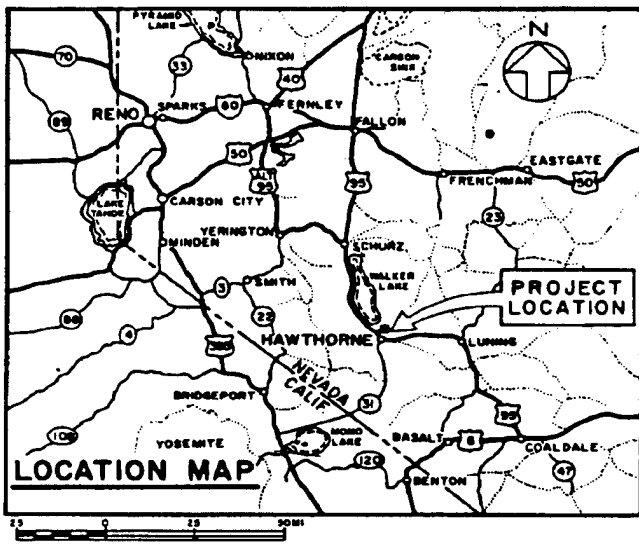


Figure 1 Location Map for Hawthorne, Nevada

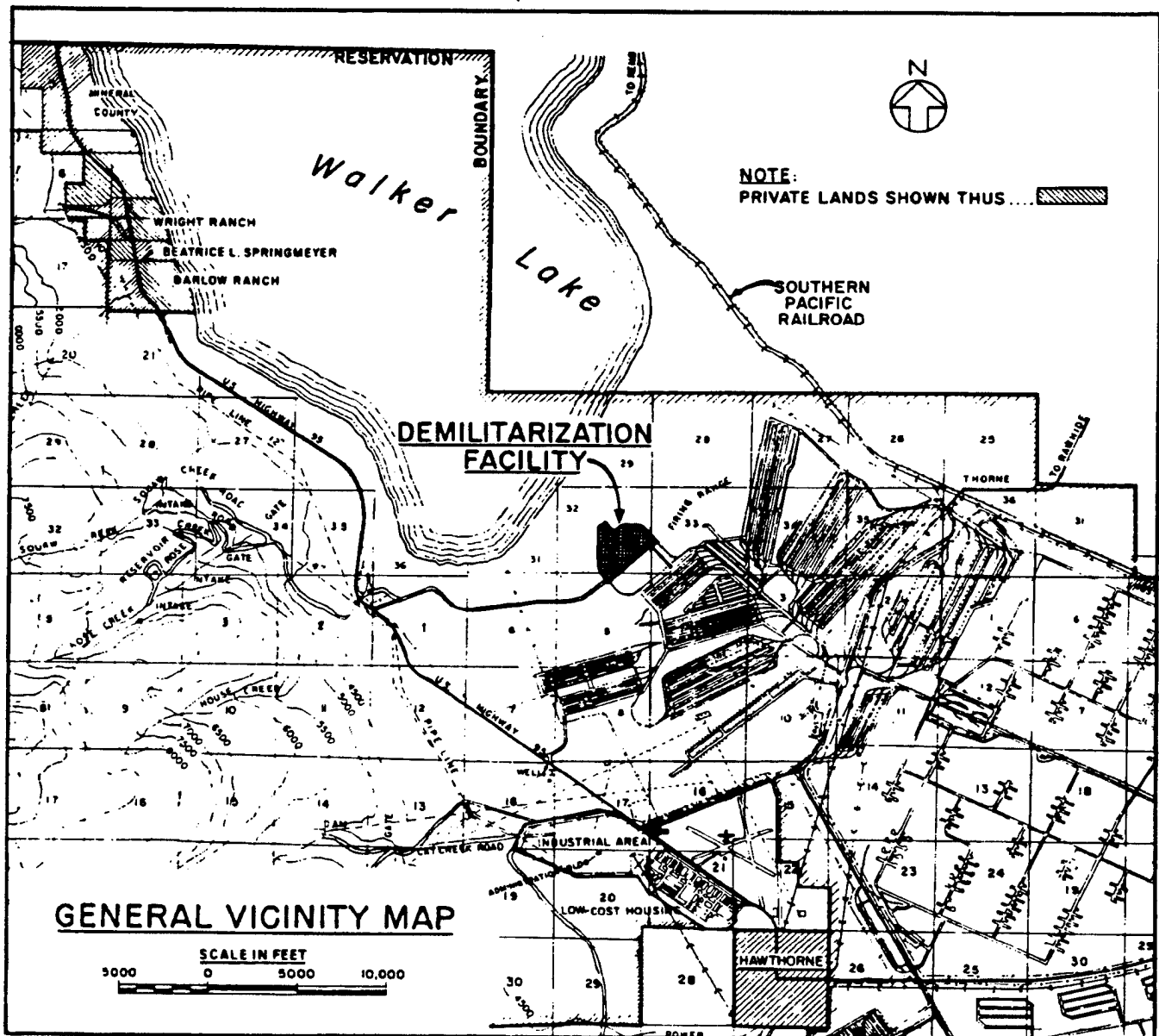


Figure 2 General Vicinity Map for Hawthorne Army Ammunition Plant showing location of WADF.

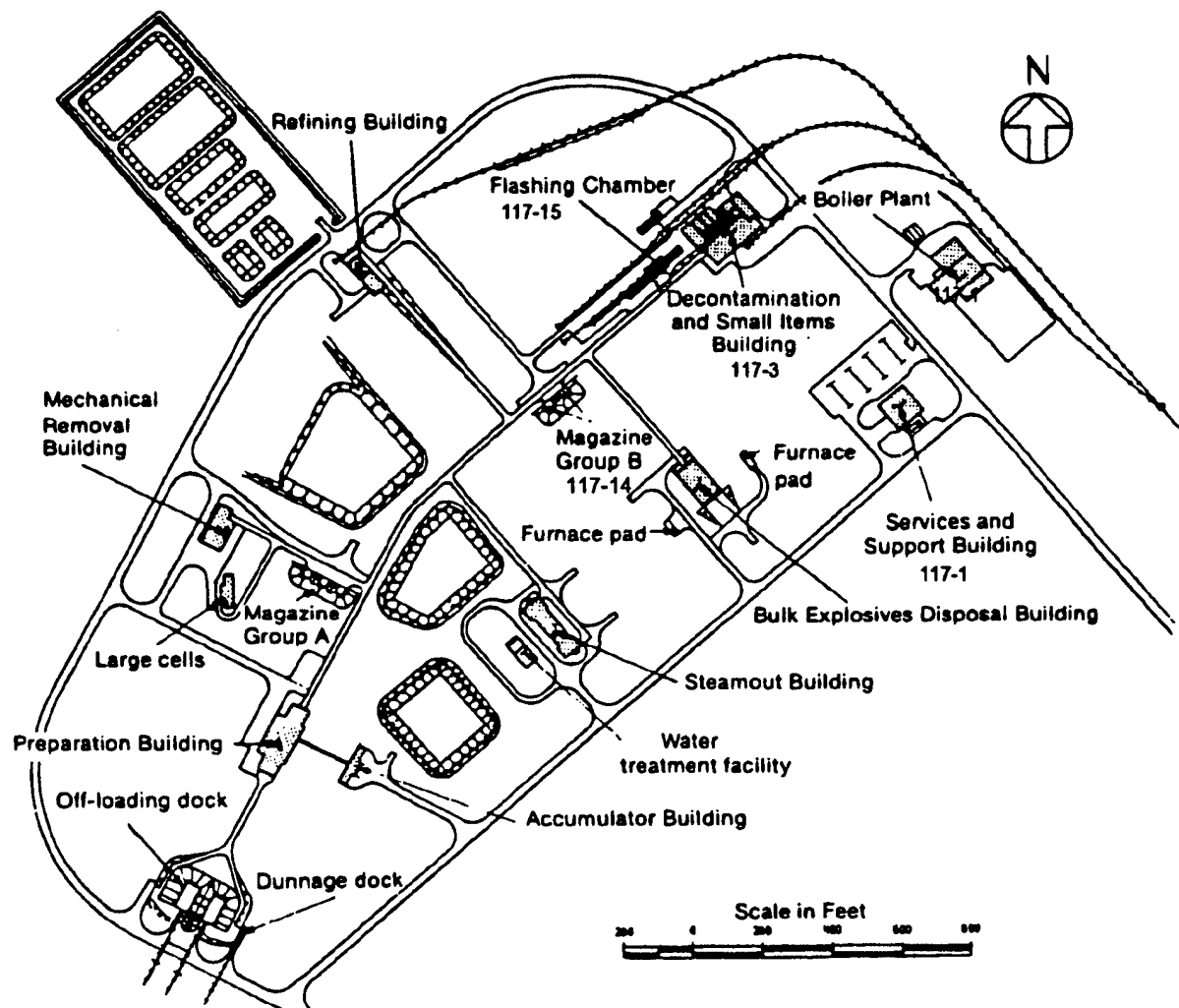


Figure 3 Overall View of the Western Area Demilitarization Facility

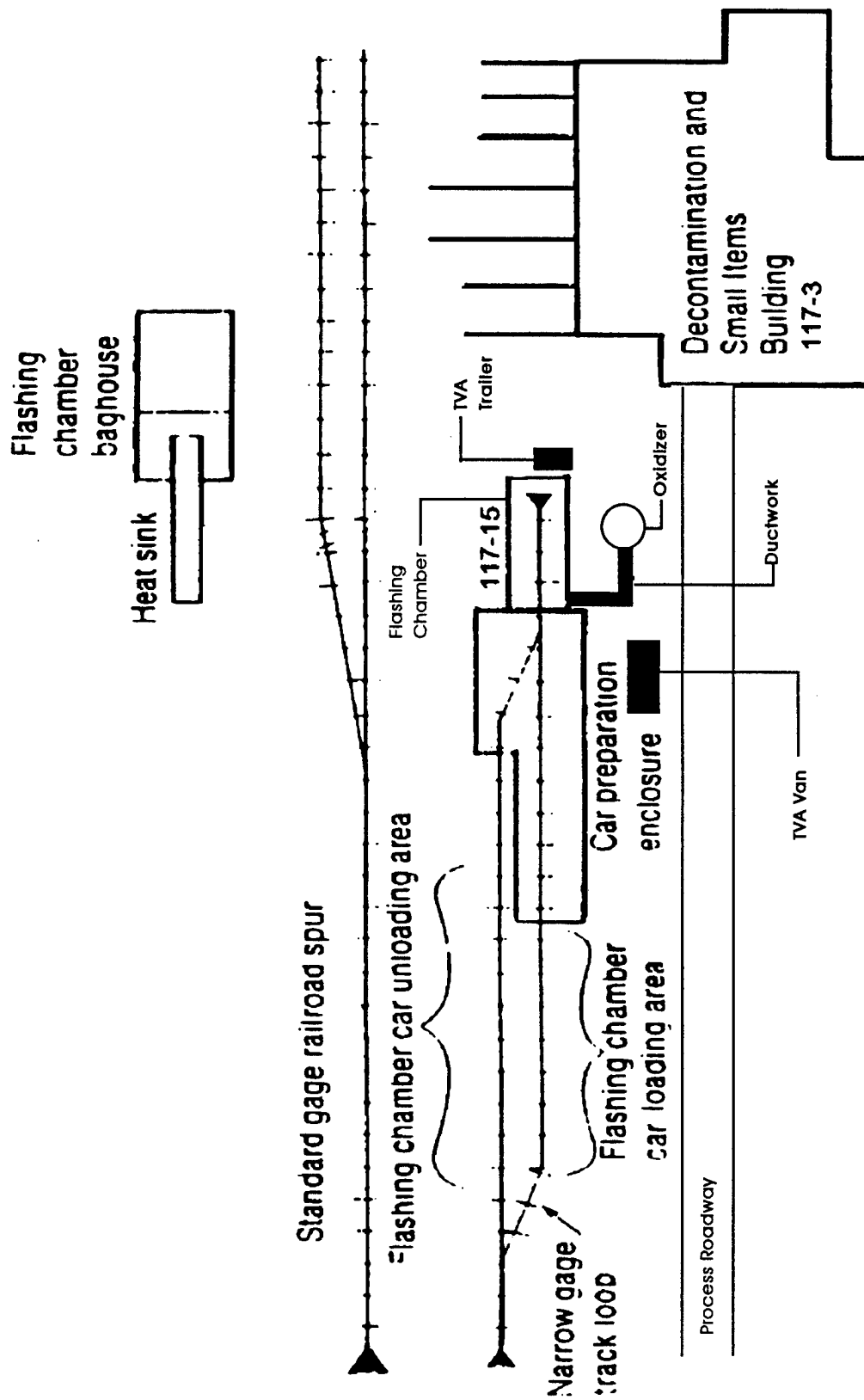


Figure 4 Arrangement of the Flashing Chamber (117-15) and Small Items Building (117-3)

The primary buildings to be used during the test are as follows:

- Building 117-1 (Service and Support) - This building contains the laboratory and will be used by TVA personnel for sample preparations and some analytical work. (The detailed analyses of each sample will be conducted at TVA laboratories in Muscle Shoals, Alabama.) This building also contains shower facilities for all personnel.
- Building 117-3 (Decontamination and Small Items Building) - Operations support area for TVA personnel. This building contains the control room for the HGD system. Break room, restrooms, and lunch areas are also in this building.
- Building 117-14 (Explosive Storage Bunker at Magazine Group B) - This facility will be used to store, weigh, and package predetermined sample size quantities of explosive material for explosive spiking. Explosive prepared for samples can be stored at this facility in nonstandard pack. Quantity of material will not exceed 20 pounds net explosive weight.
- Building 117-15 (Flash Chamber) - The testing of explosives-contaminated munitions and equipment will be done inside this facility. The flashing chamber car loading area and car preparation enclosure will be used to configure each flat car for the appropriate test. TVA will locate the control and sample van near this building.

The purpose of this test program is to test the HGD process and facility under conditions more closely simulating routine production operations of munitions demilitarization, decontamination, and disposal. It is intended to demonstrate the safety and efficacy of the process with a number of different munitions items and explosives.

## **1.2 Previous Work on HGD Concept**

### **1.2.1 Concept**

The concept of HGD is that a flow of hot gas, comprising heated air and flue gases from a burner, at a temperature as low as 500°F (260°C), can volatilize organic contaminants such as residual explosives without causing them to explode or burn. The vapors of the explosive compounds can then be directed to a thermal oxidation unit where they will be destroyed under controlled conditions. Only combustion products will exit the system. The explosives are mineralized, that is, they are converted to low molecular weight inorganic

compounds, predominantly carbon dioxide and water. A small amount of oxides of nitrogen ( $\text{NO}_x$ ) is produced from the nitrogen content of the explosives themselves but oxidizer conditions are controlled to minimize  $\text{NO}_x$  formation from combustion air. Modern explosives do not contain sulfur or chlorine compounds to contribute to emissions.

### 1.2.2 Lead Contamination

It has been noted that the MK 9 Depth Charge is weighted with lead. Metallic lead was evidently cast in place in one end of the casing and is exposed by sawing off the end of the casing for removal of the bulk explosive charge. The melting point of pure lead is 618°F. It was originally planned to limit tests with this item to a maximum of 550°F in order to avoid melting the lead. Molten lead could potentially vaporize enough lead to contaminate the chamber and generate emissions of detectable levels of lead from the oxidizer stack. This was not originally expected to be a serious problem because the MK 9 is loaded with TNT, which could probably be decontaminated alone at 500°F.

However, the presence of another non-explosive material must also be considered. Naval munitions are internally coated with a tar-like asphaltic material known as hot-melt or Flint Coat. Tests by CRREL, USAEC, and TVA indicate that explosives are soluble in the tar as it is melted during the phase of chamber heatup. On the basis of limited tests, it appears that the tar will retain an appreciable explosive content throughout a treatment cycle that would decontaminate explosive from a metal surface.

Therefore, on the basis of the present characterization of the interaction of tar and explosive, in order to ensure decontamination of items coated with tar (essentially all naval munitions, which comprise all of the present study's test items except 106mm and 175mm projectiles), the tar will have to be removed along with the explosive. That will require treatment at substantially higher temperature and longer duration. This condition caused the MK 9 to be removed from the original test sequence.

It is also considered likely that some of the munitions items to be tested will have been painted with lead paint or other protective coatings containing heavy metals. Stack gas samples will be analyzed for heavy metals such as lead, zinc, chromium, cadmium, and others as may be indicated by the specifications of the items being treated. Stack gas sampling and analysis will be conducted by U.S. Army Environmental Hygiene Agency (USAEHA).

### **1.2.3**

#### **Initial Tests**

The process was originally tested as a means for decontaminating the structure of a building in a munitions plant. Hot gas was ducted from a preheater through the building to volatilize the explosives deposited on the materials of construction (predominantly concrete block) over years of plant operation. The gas and volatilized explosives then passed out of the building to the oxidizer as described above. This initial test was conducted at Cornhusker Army Ammunition Plant near Grand Island, Nebraska, by A. D. Little Inc.

After that test was satisfactorily concluded, the preheater and oxidizer were transferred to HWAAP in 1988 and installed on the WADF flash chamber. The chamber was otherwise modified to apply the HGD process, by the installation of ducting, instrumentation and controls. A false wall was installed across the chamber to reduce the working length of the chamber. Currently, the flashing chamber can accommodate only one of the narrow gauge railcars provided to carry items for treatment.

### **1.2.4**

#### **Weston Tests**

A series of tests conducted in 1989 by Roy F. Weston, Inc., (Reference #2, RFW report) demonstrated that the HGD process could decontaminate residues of TNT from munitions and plant equipment. Ammonium picrate (Yellow D) was also decontaminated in a single trial. However, those tests were made with one or a few items at a time and the sampling and analytical procedures employed were not fully adapted to the process and its conditions. Therefore, the test results, while encouraging, were not rigorous enough to assure decontamination comparable to the traditional flashing process.

### **1.2.5**

#### **DZB Proveout**

After testing in 1989, the facility was modified. The air preheater was removed and replaced by a recirculating duct and fan so that the chamber was supplied with hot oxidizer flue gas diluted with ambient air to the desired temperature. This conserved energy compared to the original system's separately fired preheater but the system required considerable modification and adjustment to attain process temperatures of 500°F and above. The capability of the modified system to reach chamber temperatures in excess of 700°F was demonstrated in a proveout test by Day and Zimmerman/Basil Corporation (DZB), the plant contractor, in 1993 (Reference #3).

Any limitations in the HWAAP HGD system which are identified in this test demonstration program will be noted. They will be input into the generic HGD system design and technology transfer package comprising Phase II of this project (see 1.4.2 below).

### 1.3

#### **Program Participants**

##### **United States Army Environmental Center (USAEC) -**

USAEC has overall program management for the effort to demonstrate the HGD process, including project direction, coordination, and funding. USAEC also provides technical support on explosives chemistry, and specimens for methods development.

##### **Tennessee Valley Authority (TVA) -**

TVA was selected by USAEC to manage and conduct the HGD demonstration project. TVA has developed this test plan and will carry out the portions including field sampling, laboratory analysis, evaluation, and reporting. TVA will also carry out related project management activities of planning, staffing, budgeting, and scheduling.

##### **United States Army Defense Ammunition Center and School (USADACS) -**

USADACS supports preparatory work by providing explosives safety information, both for the field activities planned and for guidance in setting up the Test Plan. USADACS will lead the review of test results and will contribute to the establishment of HGD as an acceptable means of decontaminating explosive residues.

##### **Cold Regions Research and Engineering Laboratory (CRREL) -**

CRREL provides information as to explosives and munitions chemistry and characteristics to guide TVA's work on Methods Development in the areas of sampling and analysis of explosive residues.

##### **United States Army Environmental Hygiene Agency (USAEHA) -**

USAEHA has been tasked to conduct environmental sampling activities at the HGD facility. Data will be used to extend the HWAAP atmospheric emissions permit to include regular operation of the HGD facility.

##### **Hawthorne Army Ammunition Plant (HWAAP) -**

HWAAP is making the HGD system and related facilities at WADF available for testing and demonstration. HWAAP inventories of munitions on the

demilitarization account provide representative test items for decontamination. They coordinate between other organizations and the operating contractor.

**Day & Zimmerman/Basil Corporation (DZB) -**

DZB is the operating contractor for HWAAP and WADF. DZB personnel will conduct all HGD operations except for the actual sampling and analysis activities. They will spike and position test items, operate and maintain the HGD facility and equipment, and handle test items to facilitate access and sampling by TVA.

**1.4**

**Project Objectives**

**1.4.1**

**Phase I**

The objective of Phase I of the HGD Test Program is to develop data to demonstrate the efficacy and safety of the HGD process to remove and destroy residues of explosives from obsolete munitions as the final step of the demilitarization process. It comprises preliminary studies of test item spiking, sampling, and analysis. Specimens of actual contaminated munitions items will be examined in a baseline study to determine whether spiking levels used previously (in the Weston study, Reference #2) are representative of actual conditions, and whether sampling and analytical methods prescribed for artificially spiked items are effective for actual contaminants and residues. Spiking and sampling procedures will be revised as necessary to better simulate expected contamination. The contaminated items and the corresponding explosives to be tested are shown in Table 1.

**TABLE 1**  
**CONTAMINATED ITEMS AND EXPLOSIVE COMPOUNDS TO BE TESTED**

<u>ITEM</u>	<u>EXPLOSIVE</u>	<u>CONDITION</u>
1. 175mm Projectiles	Comp B (TNT + RDX)	Spiked, Demilled
2. 3"/5" Projectiles	(RDX)	Spiked
3. 106mm Projectiles	Comp A-3 (RDX)	Demilled
4. 3" Projectiles	HBX (TNT + RDX + Al)	Spiked
5. MK 54 Depth Bombs	HBX (TNT + RDX + Al)	Demilled
6. 3" Projectiles	TNT	Spiked
7. MK 25 Ship Mine	TNT	Spiked
8. 3"/5" Projectiles	Yellow D	Spiked



The test and demonstration program will examine a number of variables to determine the efficacy of HGD in decontaminating the items of current interest.

The major response variable is the effectiveness of explosives removal from the test items. It is based on the absence or presence of explosive residue after treatment, and, if present, its quantity. Other variables contributing to evaluation of the process include temperatures of the test items and the instrumented points in the system. Composition of gases discharged from the chamber and from the stack are variables to be monitored. Heatup and cooldown times for the system will be noted. The major experimental control variables that will be changed from test to test include the items treated, the type of contaminating explosive, placement of spiked or contaminated test items in the chamber, the nominal treatment temperature, and the residence time at that temperature. These variables constitute the basic description of the decontamination regimen; what is being treated to remove which contaminant, and under what conditions. Other variables will be held constant throughout the program. They include all controllable aspects of the chamber operation besides the treatment time and temperature. All tests will follow the same chamber operation with respect to air flow and chamber draft, heat delivery, and oxidizer operating parameters. Placement of the railcar with racks and total lading of items will be held constant. Only the distribution of contaminated test items among inert items included to provide thermal mass will be altered as an experimental control variable. Procedures for sampling and analysis of residues will be standardized as described in this test plan, and results will be evaluated objectively.

Some variables are independent and not subject to control or stabilization. It is likely that this category can be limited to ambient conditions of temperature and humidity, which might have some small effect on the temperature profile of the system.

A series of 31 facility demonstration tests of the HGD process will be conducted at WADF/HWAAP. An additional five operating cycles are scheduled--two to be conducted prior to actual test program operations with inert items to check temperature distribution and the effect of installing additional hot gas outlets; and three to decontaminate the chamber. See the sequence of tests in Table 3. Samples will be analyzed at TVA laboratories, with results returned to the field for ongoing process evaluation and test revision where necessary.

The most desirable demonstration of the process would call for corresponding items and explosives to be treated in spiked condition for precise measurement and in demilitarized condition to demonstrate realistic treatment requirements. However, due to current availability of the types of decontaminated items to be spiked with explosives and of washed out items to be decontaminated from a realistic demilitarized condition, the table reflects some substitutions and adjustments.

A large quantity of 175mm projectiles originally loaded with Comp B is, at this writing, being demilitarized. There are large numbers which have been furnace flashed. Therefore, spiking tests will proceed with 3"/5" projectiles which have been allocated for the purpose. However, no solvent or miscible combination of solvents has been identified which will produce a homogeneous solution of Comp A-3 suitable for spiking. Therefore, spike tests to represent Comp A-3 will be made with neat RDX. This is considered an acceptable substitution by TVA and USAEC because RDX comprises 91 percent of Comp A-3, the remainder being a non-explosive wax stabilizer.

The most nearly similar item scheduled for demilitarization soon is a trail quantity of 106mm projectiles loaded with Comp A-3. The supply of 106mm projectiles with an actual residue of Comp A-3 will be ample for verification testing of decontamination of that explosive.

There are no items normally loaded with HBX which are available decontaminated for spiked tests. Therefore, 3" projectiles will be employed for convenience in handling. There is a supply of MK 54 Depth Bomb sawed ends containing residues of HBX suitable for decontamination. Those sawed ends are heavily coated with tar-like asphaltic hot melt. Therefore, the decontamination criterion for those items, as described elsewhere, is the essentially complete volatilization of the tar.

Although TNT is a common explosive, actual munitions items originally loaded with it are not currently available. Therefore, 3" projectiles will be used for some early tests to confirm prior data. Later tests for TNT decontamination will be made with MK 25 Ship Mines, using new, never loaded casings coated with hot melt. Some will be spiked from solution in accordance with present procedures. Others were spiked for earlier work by melting a substantially larger quantity of TNT in the mine casing and rotating it for even distribution.

No items originally loaded with Yellow D are available for these tests. However, due to the large quantity of such munitions on hand for future disposal, tests will be made with 3"/5" projectiles spiked with Yellow D.

At this writing, detailed procedures for trials with ammonium picrate are still under development. The methods for spiking with, sampling from, and chemical analysis of deposits of ammonium picrate will be issued as addenda to and revisions of the appropriate section of this test plan. It is anticipated that such methods will be established and promulgated in ample time for the first test with ammonium picrate, No. 10 in the sequence in Table 3.

Progress reports and a final test report will be issued, stating the efficacy of the HGD process in decontaminating munitions retaining explosive residues. Recommendations for implementation and advantages and limitations of the process will be described for the items tested. Assuming that the HGD process has been satisfactorily demonstrated, the program results and recommendations will be submitted to appropriate authorities in the explosives safety community. When the safety and efficacy of the process has been accepted, the facility will be put into production at HWAAP. It will decontaminate all appropriate items for unrestricted disposal. TVA does not anticipate being the lead agency in that process but will, of course, consult as required. Any additional recommendations or requirements that are developed will be incorporated into the technology transfer package, outlined below.

#### 1.4.2 Phase II

The objective of Phase II of the HGD program, not otherwise discussed here, is to provide for technology transfer. A generic design for construction of a new HGD facility will be prepared. It will incorporate results and recommendations from tests performed at HWAAP to establish nominal operating conditions and procedures. The facility design will incorporate industrial practice to help ensure the following: 1) efficient thermal processing in the temperature range determined to be effective, 2) oxidative destruction of volatilized explosives with appropriate emission controls. Facility design will incorporate provisions for safe handling and processing of explosive contaminated items. Established procedures for safe handling of the contaminated items will be accommodated. The HGD system will process the items under treatment conditions determined to be adequate for effective decontamination. The design of the facility will minimize the likelihood of accumulation of redeposited explosives on facility surfaces, a contingency which will be inspected for under this test plan.

## **1.5**

### **Study Conditions**

#### **1.5.1**

##### **Facility Capabilities**

During the initial facility tests by Weston, it was found that a temperature of 500°F, maintained for six hours, would effectively decontaminate surface deposits of TNT from the munitions and plant process items studied at that time. The effect of higher temperatures could not be established because the HGD system, as configured at that time, took longer than six hours to increase chamber temperature from 500°F to 600°F. It was therefore presumed that the TNT present would have been volatilized before the temperature reached 600°F. Ammonium picrate was decontaminated in a single test at 600°F sustained for 48 hours. It is not known whether such a long period of treatment was necessary but it was considered desirable at the time to minimize the chance that some ammonium picrate might partially degrade to picric acid, which is considered to be more sensitive to detonation and therefore more hazardous.

After conversion to a recirculating heating system the HGD facility at HWAAP was demonstrated in the DZB's recent proveout test (Reference #3) to have the capability of attaining nominal operating temperatures in excess of 700°F. A temperature of 800°F was reached in testing but could not be sustained for the full period scheduled.

Temperature distribution in the chamber during those trials was relatively uneven, especially during heatup of the chamber. This was likely due to the geometry of the chamber, cold air in-leakage, and the orientation of the test items which did not allow space for gas circulation. The data from Tests A and B with inert items will be considered with a view towards relocating the temperature control element to the area in the chamber which is the slowest to reach the desired temperature. The temperature element will remain fixed for all tests with explosives. If temperature distribution is uneven enough to call for further remedy, it will be made as an increase or offset in temperature setpoint instead of relocating the control element again. This will provide for the planned treatment of items in that area. Objects in other areas of the chamber will likely be processed at a higher effective temperature for a longer period than the nominal value for the test.

#### **1.5.2**

##### **Projected Requirements**

The present facility configuration allows for more rapid heatup of the HGD chamber than when a separate preheater was employed. A nominal operating temperature of 500°F can be reached more rapidly and efficiently and treatment

temperatures greater than 500°F can also reasonably be tested. In the present program, tests will start at the conditions of 500°F for six hours employed in the Weston report (Reference #2) in the case of explosives compounded with TNT and RDX. It is provisionally assumed that such treatment will remain effective for TNT residues with the different items, chamber loadings, and sampling/analytical criteria to be applied. It is also assumed that such conditions are likely to be effective in decontaminating RDX as the main constituent of Comp A-3 and as an ingredient together with TNT in Comp B and HBX. If required for effective decontamination of different explosives or larger quantities, treatment conditions will be increased up to the maximum readily attainable chamber temperature, approximately 700°F, and the longest duration considered to be operationally feasible, a time yet to be determined. Tests with ammonium picrate will begin with longer duration as a safety factor.

#### **1.6 Test Schedule**

The schedule for the HGD facility test program is shown as a Gantt chart in Figure 5. It projects that laboratory development and baseline studies will take place in March and April 1994 and that facility tests will begin in June and last for about 120 days as shown in Figure 6. For the purposes of scheduling, it was assumed that each facility test at HWAAP would take three days. The allowance for facility maintenance (approximately 3-4 days) is shown in the chart in conjunction with Tests C, D, and E. The current test plan calls for working ten hours per day, seven days per week. Data evaluation and report preparation will complete the test program in January 1995. During tests C and D, TVA personnel will rotate to Muscle Shoals, Alabama, for a five-day break.

#### **1.7 Health and Safety**

TVA field and laboratory personnel attended a two-day Explosives Safety Course provided by the U.S. Army Defense Ammunition Center and School (USADACS) during March 1994.

During test operations safe practices described in the Health and Safety Plan (Appendix C) will be followed. The HGD facility portion of that plan is based on the HWAAP Accident Prevention Plan. This will insure that all personnel will follow the same guidelines. The laboratory protocol (Appendix A) will direct laboratory personnel at TVA to follow appropriate practices for handling samples for analysis, based on their established practices for hazardous and toxic materials and the specific requirements of the explosives being analyzed. Methods and Procedures (Appendix B), Field Sampling, will direct TVA field personnel to follow

PROJECT: HOT GAS DECON TEST  
 MANAGER: R A ALMOND  
 AS OF DATE: 11/01/93

PROJECT SCHEDULE

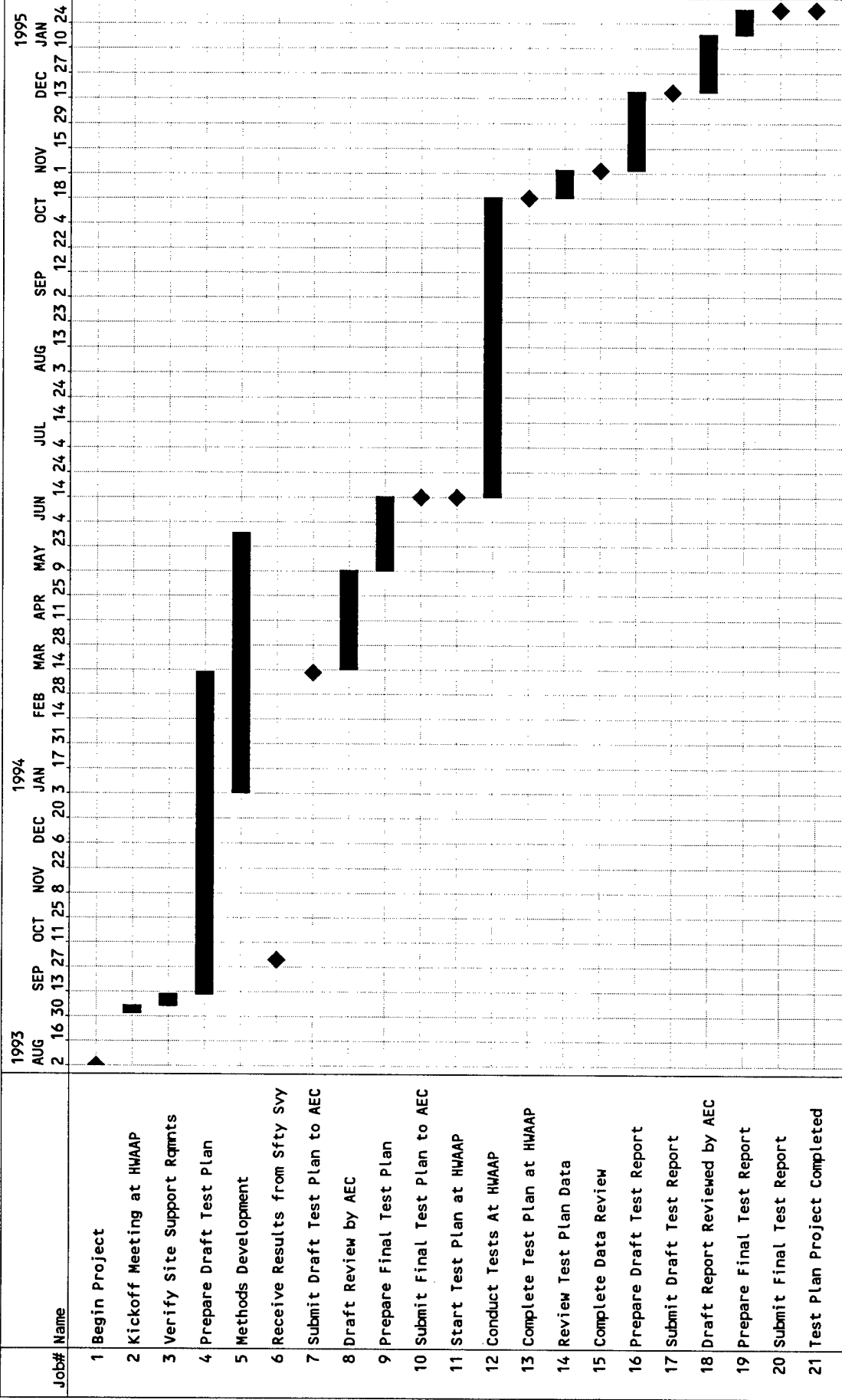


Figure 5 HGD Test Plan - Project Schedule

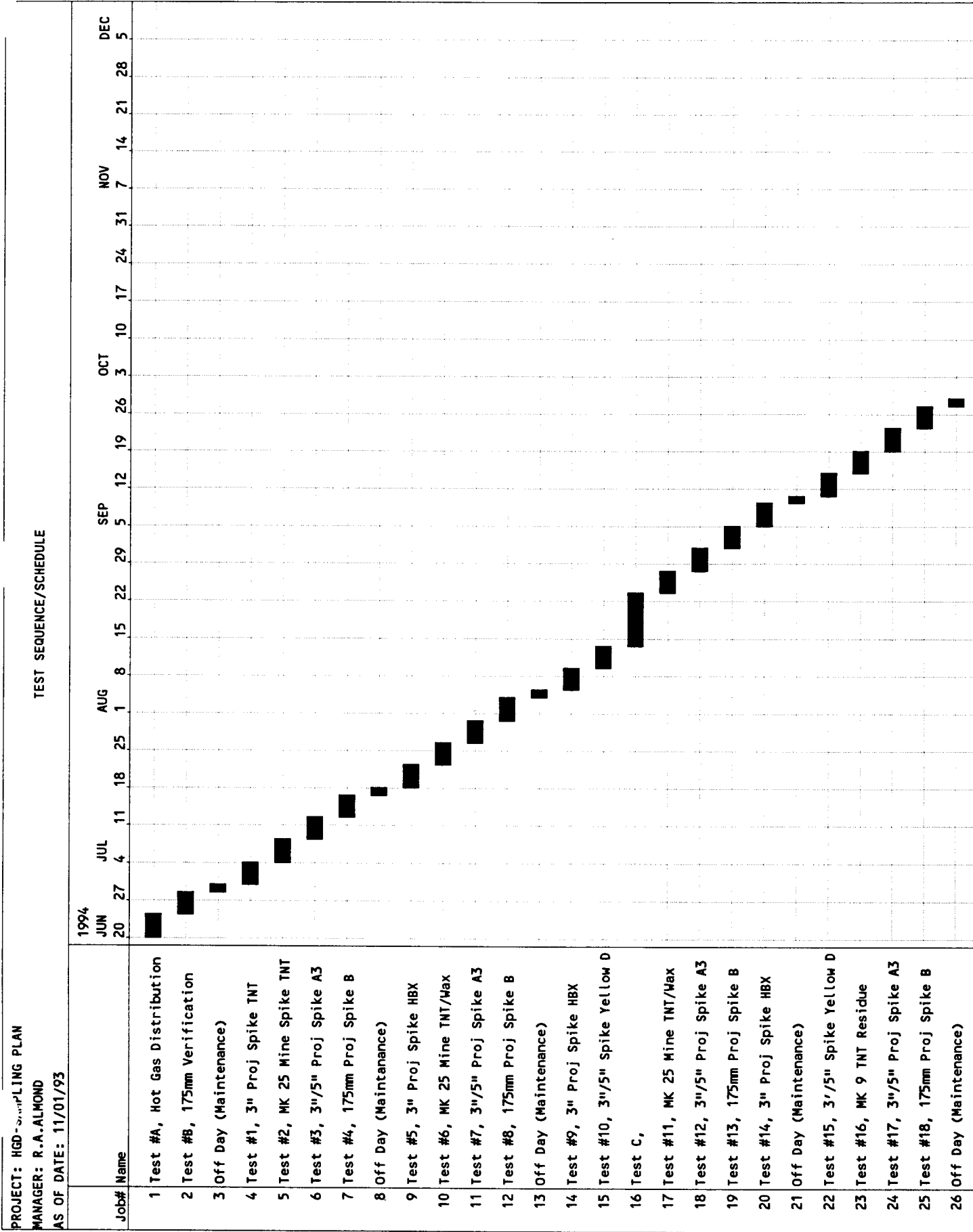


Figure 6 HGD Test Plan - Test Sequence/Schedule

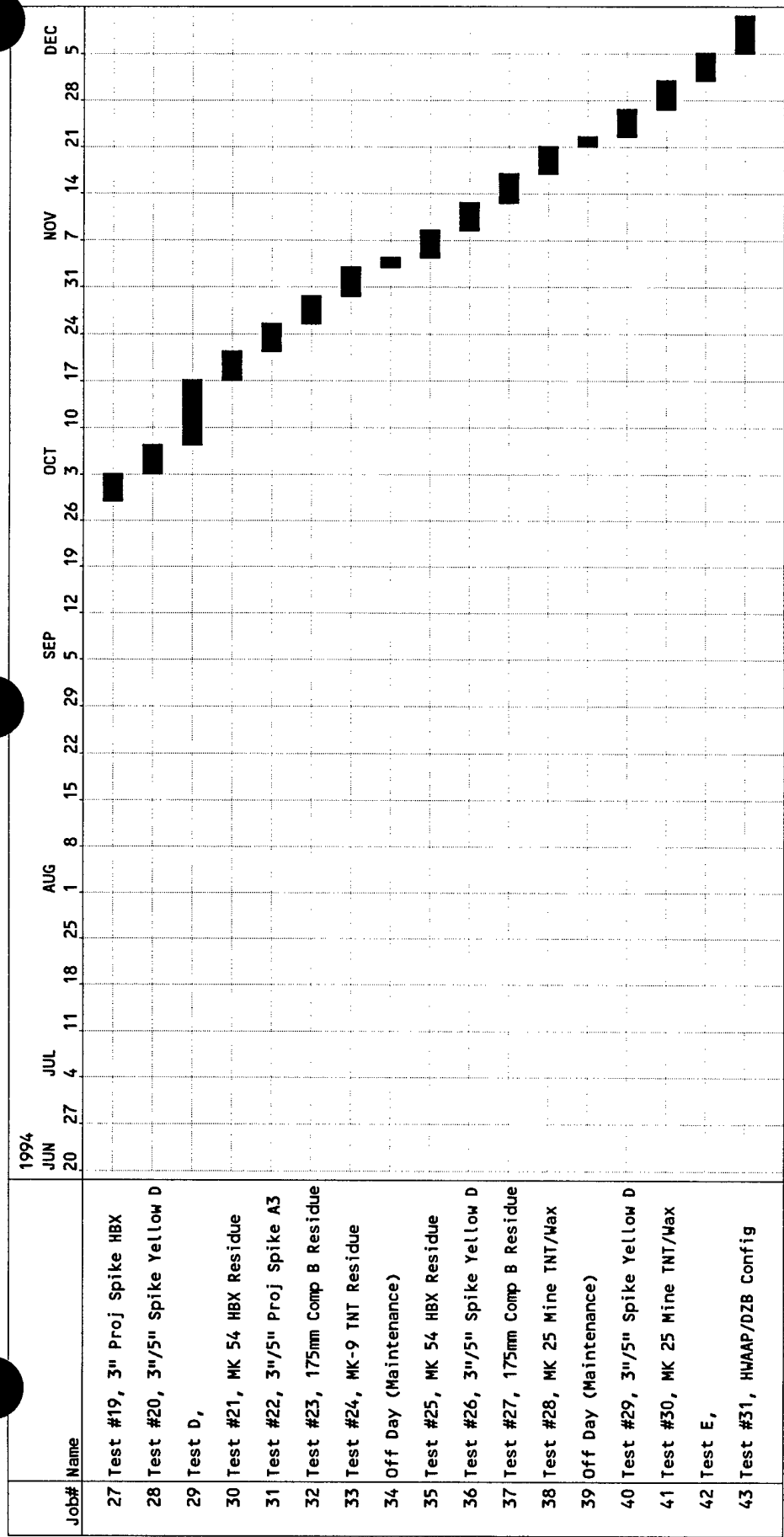


Figure 6, continued



appropriate practices for collecting samples for analysis, based on their established practices for hazardous and toxic materials and the specific requirements of the explosive being sampled.

Test activity responsibility assignments will also contribute to safety. All handling of explosives in quantity will be performed by DZB personnel, previously trained and experienced in explosives operations. DZB will spike the test items and load them (and the contaminated items to be tested later) into fixtures, load the fixtures onto railcars, move the cars into and out of the HGD chamber, and handle the treated items for sampling access. TVA field personnel will handle explosives only in the amounts being recovered by sampling individual items by solvent flush and wipe procedures. TVA laboratory personnel will handle explosives in the amounts required to determine spiking levels and in lesser amounts to calibrate and operate analytical equipment. All TVA personnel in both locations will employ the proper techniques from USADACS training.

## **2.0 METHODS DEVELOPMENT**

The general objectives and context of methods development to be performed by TVA are as follows. A detailed laboratory plan in accordance with EPA Level III is given in Appendix B.

### **2.1. Laboratory Studies - Analytical Procedure Refinement and Confirmation**

A TVA laboratory will be prepared to handle samples derived from HGD tests. Sample preparation and analytical equipment and instrumentation will be set up. Safety measures will be promulgated and applied.

The Weston tests applied Method 8330 for the determination of TNT and its coproducts to the analysis of flush and wipe samples taken from HGD test items. Method 8330 was developed for the purpose of determining the explosive contamination level of soils and water. The actual applicability of Method 8330 to HGD samples will be investigated and the procedures will be further modified as required to give accurate and consistent results. The same method also yields analytical results for RDX and a number of TNT and RDX byproducts and coproducts, as well as a number of other explosives not of present interest.

Ammonium picrate, however, is analyzed by a separate procedure. That procedure will be similarly studied and refined to suit the characteristics of the samples to be obtained during HGD testing of that explosive.

In the Weston tests the most commonly used solvent for test item spiking, sampling, and analytical extractions was acetone. There was some use made of acetonitrile. Those solvents will be reviewed and alternatives will be evaluated for suitability. Chemical, operational, and safety performance will be considered.

These procedures and the corresponding activities for spiking and sampling are shown in Appendix E. They are being handled as laboratory control documents with all revisions tracked and recorded. The latest versions of these documents will be issued as standard operating procedures at the commencement of testing. Necessary revisions will be issued to field and laboratory staff. The final versions will be presented in the test report.

### **2.2 Field Baseline Studies**

Prior to commencement of HGD facility testing, field baseline studies will be made at HWAAP. The objectives of those studies are as follows:

### **2.2.1      Spiking and Sampling Procedures**

Spiking and sampling procedures have been prepared by the TVA laboratory. In general, the explosive of interest will be deposited in or on the test item by evaporation of solvent from a liquid solution of explosive in acetonitrile or other suitable solvent. Sampling of the spiked items will be performed by solvent removal of the surface deposit spike. The procedures are described in detail in Appendix E.

### **2.2.2      Analysis**

Baseline study samples taken by solvent flushing and solvent wiping will be air-freighted to the TVA laboratory, as is planned for facility testing. They will be analyzed by the appropriate method established in the laboratory studies described above and as shown in Appendix B. Quality control samples will be included in the initial field studies by TVA field personnel. They will include, but not be limited to, such measures as blank samples of solvent, samples directly spiked with known levels of explosive, and duplicates.

Analytical results will be reviewed in the laboratory for several data quality indicators. Chromatograms will be reviewed for indications of the presence of interfering compounds. Surrogate recovery will be reviewed to ensure it falls between 80 and 120 percent. Quality control sample recovery for samples introduced by the laboratory in the course of routine operations will be reviewed to ensure it falls between 80 and 120 percent. Matrix spikes and matrix spike duplicates will be reviewed to ensure they fall between 75 and 125 percent recovery. Blanks will be reviewed to ensure no carryover or contamination is indicated.

Field duplicates, field quality control unknowns, and field blanks will be reviewed to ensure recovery falls within similar limits and that there is no indication of carryover or contamination.

Experimental data will be reviewed for consistency. For example, recovery of spiked compound from solvent washes and smear tests should be 100 percent within experimental uncertainty for some experiments described herein. Distribution of explosives among successive washes or within one wash as a function of extraction time should be logical and consistent with known physical and chemical processes.

### **2.2.3 Contamination Level**

After field trials of the spiking and sampling procedures, a selection of demilitarized but not yet decontaminated items will be sampled for analysis. The same methods will be employed, with any necessary revisions by TVA. The objective will be to determine the typical level of explosive contamination in actual munitions from upstream processing. A further objective will be to determine the effect of actual explosive residue upon sampling and analysis procedures and effectiveness. Impurities and physical conditions might have an effect to be considered.

### **2.2.4 Spiking Review**

If the typical degree of actual contamination in demilitarized items differ significantly from the 10 grams of explosive per item employed in spiking, the level of spiking will be adjusted to better simulate the amount of actual residue to be expected.

## **2.3 Experimental Plan**

The methods development task of the HGD project will be conducted in TVA's analytical and research laboratories at Muscle Shoals, Alabama. In this task, analytical equipment will be procured and configured, analytical procedures will be defined and written, explosive extraction procedures will be tested on spiked surfaces, a bench scale testing process will be devised to volatilize the target compounds, and all other laboratory or test procedures will be developed as needed to facilitate the completion of the test plan.

### **2.3.1 Chemical Compounds Applicable to the Experimental Plan**

Fifteen chemical compounds (listed in Table 2) have been identified that have a potential to be present in the test samples for analysis, and thus are relevant to the development of analytical and test methods. These compounds include both the primary explosive compounds plus those compounds that may appear in the samples for other reasons. The list of other compounds includes (1) degradation products of the primary compounds, (2) manufacturing by-products or impurities present in the original explosive, and (3) candidate surrogate compounds, which, if found to be suitable, will be added to the samples by laboratory personnel to verify proper functioning of the analytical equipment.

The effects of two other materials not characterized as explosives or even as distinct chemical compounds will be taken into consideration. Wax is employed in composition explosives A-3, and HBX as a desensitizer and physical property

modifier. Hot melt, a tar or asphaltic material, is commonly employed to seal metal surfaces away from direct contact with explosives, especially in naval munitions. The effects of these materials upon the decontamination process, sampling, and chemical analysis are yet to be established.

**TABLE 2**  
**EXPLOSIVES AND POTENTIAL DEGRADATION PRODUCTS**

<u>Chemical Name</u>	<u>Abbreviation</u>
<u>Explosive Compounds</u>	
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
2,4,6-Trinitrotoluene	TNT
Ammonium picrate	Yellow-D
<u>Degradation Compounds</u>	
Methyl-2,4,6-trinitrophenylnitramine	tetryl
Octrahydro-1,3,5,7-tetranitro - 1,3,5,7-tetrazocine	HMX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Nitrobenzene	NB
4-Amino-2,6-dinitrotoluene	4-Am-DNT
2-Amino-4,6-dinitrotoluene	2-Am-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

### 2.3.2 Sub-Projects of the Experimental Plan

The specific sub-projects to be undertaken by the TVA laboratories during the methods development task are:

- Setting up, configuring, and verifying EPA Method 8330 (revision 0, November 1992) to ensure it can be run properly by the TVA laboratories.

- Developing and testing a method to analyze for ammonium picrate (in the form of picric acid) using high performance liquid chromatography.
- Determining what the detection limits for both EPA Method 8330 and ammonium picrate methods are. A target detection limit of 0.5 ppm for all compounds was set at a conference among USAEC, TVA, and USADACS 4-5 January 1994. It was agreed that 0.5 ppm represents a reasonable analytical effort.
- Developing a spiking method for both projectile-type items and large munition items.
- Developing a suitable solvent extraction procedure for use with projectile-type items (i.e., those items which are sufficiently small and enclosed such that a modest volume of solvent can be used to fully rinse the interior surface).
- Developing a wipe extraction method for sampling metal surfaces (e.g., spiked areas of large munition items or HGD chamber surfaces) to determine the level of explosive contamination.
- Devising a suitable sampling train to analyze the off-gas leaving the HGD chamber. The train is to be based on the EPA's Modified Method 5 (MM5). This will include testing of the capability of various solvents and resins to absorb the target compounds in question.
- Verifying whether volatile organics (i.e., various decomposition products from the decontamination process) may be determined from the sampling train in addition to the target compounds mentioned above.
- Investigating the problems which are likely to arise in obtaining accurate analyses of the target compounds on asphalt-coated surfaces. Here, the possibility of a multitude of interfering organic compounds or solubility of the target compounds in hot asphalt may preclude success.

Where appropriate, the results of each of the sub-projects will be written into step-by-step procedures for use in the laboratory or in the field.

The brief discussions below summarize the work activities and experimental objectives to be undertaken during the methods development phase.

Appendices A and B contain additional details of the formal laboratory procedures to be adopted during the experimental work.

- Utilizing current knowledge of liquid chromatography as to the effects of various solvents, flow rates, pressures and other system parameters, TVA laboratories will configure and modify Method 8330 and its associated extraction, preparation, and cleanup procedures to run as effectively as possible and to meet the required 0.5 parts per million detection limit.
- Beginning with the established chromatography method, "Ammonium Picrate in Water and Wipe Samples by High Performance Liquid Chromatography" (referenced in Appendix B) as a starting point, the TVA laboratories will make modifications and experiments to determine the proper chromatograph operating conditions needed to analyze samples for ammonium picrate. Expected performance should be similar to Method 8330 with regard to precision and sensitivity.
- While analytical procedures are being established, a variety of surrogate compounds will be tested in order to identify the surrogates best suited to the analytical method for each explosive type.
- Tests will be made to determine a wash-type solvent extraction method for projectiles that is suitable for extracting explosives present down to very low mass levels. Experiments will be conducted to determine the number of rinses and the volume of each rinse needed to achieve a 99 percent or greater recovery of the explosive. The TVA laboratories will spike projectiles with target compounds in mass levels ranging from 10 grams, the spike level to be used in the HGD chamber tests, down to a few milligrams, with the minimum level depending on what is learned in the work as it is conducted.

As an additional test objective, tests will be conducted to define the minimum volume of solvent needed to extract a high and repeatable percentage of the explosive material in a single wash. A single wash with a minimum volume is desirable so as to maximize the concentration of the explosive in solution for analytical purposes, and thereby lower the mass level that is detectable in the projectile. If the extraction percentage is repeatable within a few percentage points, a single extraction can be directly used to estimate explosive quantities.

- Utilizing knowledge from the extraction process and knowledge of detection limits, suitable wipe tests will be devised which will quantitatively remove target compounds from a surface on which they have been deposited. A similar approach to maximize the explosive concentration in the wipe will be applied to the development of the wipe test method in order to enhance the detection sensitivity of the method.
- TVA laboratories will design a tube furnace system to volatilize spiked target compounds in order to facilitate testing of the off-gas sampling train. The furnace should be able to reach temperatures high enough to simulate the hot gas decontamination equipment in the field. Utilizing knowledge about solubilities and absorbencies of the target compounds and their decomposition products, various absorbing compounds and solutions will be tested in the sampling train.

Additional questions to be answered in the tube furnace work are: Will other volatile organic compounds (pollutants) be detected in the sampling train if they are present? If they are present, will they interfere with the methods for the target compounds?

- Asphalt used to coat the interior of Naval munitions will be spiked with target compounds. Then, various heating regimes will be examined, each followed by extraction washes to determine if the target compounds can be removed from the asphalt. Chromatograms will be examined to determine whether decomposition products interfere with the analysis of the target compounds. Results of investigation of asphaltic hot melt under way at CRREL will be reviewed to avoid duplication of effort and to gain maximum benefit from laboratory testing.
- Ammonium picrate will be tested in the tube furnace in TVA's laboratory. A main objective, in addition to qualifying the gas sampling system as described above, is to determine a suitable treatment temperature that will completely volatilize the ammonium picrate but will not evolve ammonia to leave a residue of picric acid. Effort will also be made to ascertain the necessary retention time to complete the removal of ammonium picrate. Considering the small scale of the experiment, this will probably be measured by a proportionate comparison of the retention times needed for volatilization of ammonium picrate and TNT.



Safety. Before starting the laboratory work, laboratory personnel will be trained in the proper and safe handling procedures for small quantities of explosives.

### **3.0 HOT GAS DECONTAMINATION TEST PROGRAM AT HWAAP**

#### **3.1**

##### **Rationale**

As previously described, the objective of the test program is to demonstrate the efficacy and safety of the HGD process toward decontamination of munition items containing small residues of explosives after demilitarization. Prior testing demonstrated the applicability of the process and the HWAAP facility to decontaminate residues of TNT from various items treated singly or in small lots. The rationale of the present test program is to extend the process to additional different items and explosives and to test them in quantities approaching the projected usage of the facility in routine operations. As will be seen below, the items to be treated will be loaded into holding racks or arranged on wire pallets on the chamber railcar. The racks employed will position the items on the railcar with adequate separation to allow for circulation of the hot gas flow through the chamber. This will maximize heat transfer to the items and mass transfer of vaporized explosive compounds away from the items and from railcar, racks, and chamber surfaces where they might be redeposited.

Although this test program will test different items, different explosives, and will treat a greater number of items per test than in previous work; the railcar will seldom be fully loaded in the interest of maintaining a manageable number of test specimens and samples. In an operational mode for the facility it will be desirable to treat as many items per batch as possible to enhance HGD and overall WADF productivity.

In most cases, the quantity of items treated could likely be increased by closer spacing of holding racks and vertical stacking of racks up to the height of the HGD chamber door. The car lading planned for tests of 175mm projectiles approaches the rated weight capacity of the railcar, although some overloading of the railcar might be acceptable in light of the short distances to be travelled. Decontamination would not be expected to be adversely affected by such measures, possibly requiring only a few confirmatory tests to prepare the facility for operational use.

Other measures that have been mentioned to increase HGD throughput, such as palletization, random filling of expanded metal containers, or simple stacking of items would place items in contact with each other and the railcar bed or chamber floor. One trial of such a high capacity lading of the chamber is scheduled as Test #31 at the conclusion of this series. Selection of a particular

item and exact configuration of Test #31 will be determined by HWAAP/DZB to reflect installation priorities and preferences. Further trials of high capacity lading will be conducted as project resources permit. The objective is to match decontamination throughput to other steps in the demilitarization process.

### 3.2 Test Sequence

The sequence of tests is shown in Table 3. A complete set of Test Sequence Diagrams showing item configuration is contained in Appendix D. Two preliminary tests (A and B) with inert items are intended to demonstrate the effectiveness of changes made in hot gas distribution and in item arrangement on the railcar. Three other treatment cycles are included as Tests C, D, and E, in which the chamber will be heated to its maximum sustainable temperature for 24 hours to ensure complete decontamination of the facility itself. This is a precautionary measure taken in addition to any similar surface decontamination shown to be necessary by the test program. The need for such precaution is indicated by the construction of the chamber which has open seams in the interior cladding, with the possibility of explosives or derivative compounds penetrating into chamber insulation. There are to be 31 facility tests with explosives present. Testing will begin with 3" projectiles spiked with TNT. Treatment conditions, 500°F for six hours, are as established by the Weston tests. TNT will be tested first to confirm prior data (Reference #2) and to start work with the explosive for which there are data and experience to draw on. As the test sequence progresses, other items and explosives will be processed. Initial treatment conditions for munitions that had been loaded with composition explosives containing TNT and/or RDX will be the same as for TNT. RDX is expected to be volatilized and probably decomposed at the same temperature-time relationship as TNT. Items containing Yellow D (Ammonium Picrate) will be treated at a higher temperature for a longer time, provisionally 600°F for 12 hours, in order to minimize the risk that ammonium picrate would be partially decomposed by volatilization of ammonia, leaving a residue of the more sensitive picric acid. The severity of those conditions may be reduced if initial results indicate that it may be safely done. Of course, detection of a residue of any explosive after HGD will call for the next test with that explosive to be made at more severe treatment conditions of temperature and/or retention time.

It has been noted that the MK 9 Depth Charge is weighted with lead. Metallic lead was evidently cast in place in one end of the casing and is exposed by sawing off the end of the casing for removal of the bulk explosive charge. The melting point of pure lead is 618°F. It was originally planned to limit tests with

**TABLE 3  
TEST SEQUENCE**

Test No	Test Item	Item Source	Test Status		Test Conditions	
			Explosive	Condition	Time	Temp. (F)
A	Proveout conditions – Test Discontinued, configuration dismantled.					
B	Inert 175mm projectiles (in racks) to verify heat distribution					
1	3" Projectiles	FF – 13	TNT	Spiked	6 Hrs	500
2	3"/5" Projectiles	FF – 13	RDX	Spiked	6 Hrs	500
3	175mm Projectiles	FF – 13	Comp B	Spiked	6 Hrs	500
4	3" Projectiles	FF – 13	TNT	Spiked	6 Hrs	550
5	3" Projectiles	FF – 13	HBX	Spiked	6 Hrs	500
6	MK 25 Ship Mines	Unused– hot melt coating	TNT	Spiked	16 Hrs	750
7	3"/5" Projectiles	FF – 13	RDX	Spiked	Time/Temp. based on results #2	
8	175mm Projectiles	FF – 13	Comp B	Spiked	Time/Temp. based on results #3	
9	3" Projectiles	FF – 13	HBX	Spiked	Time/Temp. based on results #4	
10	3"/5" Projectiles	FF – 13	Yellow D	Spiked	12 Hrs	600
C	Operate system at maximum sustainable temperature for 24 hours with chamber empty.					
11	MK 25 Ship Mines	Unused– hot melt coating	TNT	Spiked	Time/Temp. based on results #6	
12	3"/5" Projectiles	FF – 13	RDX	Spiked	Time/Temp. based on results #7	
13	175mm Projectiles	FF – 13	Comp B	Spiked	Time/Temp. based on results #8	
14	3" Projectiles	FF – 13	HBX	Spiked	Time/Temp. based on results #9	
15	3"/5" Projectiles	FF – 13	Yellow D	Spiked	Time/Temp. based on results #10	
16	MK 54 Depth Bombs	Sawed End (Demil)	HBX	Residue	16 Hrs	750
17	3"/5" Projectiles	FF – 13	RDX	Spiked	Time/Temp. based on results #12	
18	175mm Projectiles	FF – 13	Comp B	Spiked	Time/Temp. based on results #13	
19	MK 54 Depth Bombs	Sawed End (Demil)	HBX	Residue	Time/Temp. based on results #16	
20	3"/5" Projectiles	FF – 13	Yellow D	Spiked	Time/Temp. based on results #15	
D	Operate system at maximum sustainable temperature for 24 hours with chamber empty.					
21	MK 54 Depth Bombs	Sawed End (Demil)	HBX	Residue	Time/Temp. based on results #19	
22	106mm Projectiles	Demil Facility	Comp A – 3	Residue	Time/Temp. based on results #17	
23	175mm Projectiles	Demil Facility	Comp B	Residue	Time/Temp. based on results #18	
24	3"/5" Projectiles	FF – 13	Yellow D	Spiked	Time/Temp. based on results #20	
25	MK 54 Depth Bombs	Sawed End (Demil)	HBX	Residue	Time/Temp. based on results #21	
26	MK 25 Ship Mines	Unused– hot melt coating– Mines from previous testing	TNT	Old Spike	Time/Temp. based on results #11	
27	175mm Projectiles	Demil Facility	Comp B	Residue	Time/Temp. based on results #23	
28	106mm Projectiles	Demil Facility	Comp A – 3	Residue	Time/Temp. based on results #22	
29	3"/5" Projectiles	FF – 13	Yellow D	Spiked	Time/Temp. based on results #24	
30	MK 25 Ship Mines	Unused– hot melt coating– Mines from previous testing	TNT	Old Spike	Time/Temp. based on results #26	
E	Operate system at maximum sustainable temperature for 24 hours with chamber empty.					
31	Operate system with items and configuration as specified by HWAAP/DZB.					
	Additional tests to be conducted as time and resources will allow.					

this item to a maximum melting point of 550°F in order to avoid melting the lead. Molten lead could potentially vaporize enough lead to contaminate the chamber and generate emissions of detectable levels of lead from the oxidizer stack. This was not originally expected to be a serious problem because the MK 9 is loaded with TNT, which could probably be decontaminated alone at 500°F.

However, the presence of another non-explosive material must also be considered. Naval munitions are internally coated with a tar-like asphaltic material known as hot-melt or Flint Coat. Tests by CRREL, USAEC, and TVA indicate that explosives are soluble in the tar as it is melted during the phase of chamber heatup. On the basis of limited tests, it appears that the tar will retain an appreciable explosive content throughout a treatment cycle that would decontaminate explosive from a metal surface.

Therefore, on the basis of the present characterization of the interaction of tar and explosive, in order to ensure decontamination of items coated with tar (essentially all naval munitions, which comprise all of the present study's test items except 106mm and 175mm projectiles), the tar will have to be removed along with the explosive. That will require treatment at substantially higher temperature and longer duration.

In small-scale tests a typical treatment of 750°F for 16 hours was required to remove 99 percent of the tar, leaving only an ashy residue which is considered suitable for solvent extraction and analysis to demonstrate removal of explosives. Lower temperatures took much longer to volatilize the tar, 700°F for 32 hours. A higher temperature of 800°F vaporized over 99 percent of the tar in the lab furnace in only four hours. Heatup time will likely govern the most efficient treatment in the HGD facility.

At the conclusion of HGD treatment of items containing hot melt coating, they will be visually inspected. Any item containing a film or deposit of tar will be considered not decontaminated because the presence or absence of explosive could not be reliably determined with present methods. An item with only a light residue of loose ashy material will be sampled by washing or wiping with solvent. Chemical analysis will be performed to determine whether any explosive remained after volatilization of the tar.

It is also considered likely that some of the munitions items to be tested will have been painted with lead paint or other protective coatings containing heavy metals. Stack gas samples will be analyzed for heavy metals such as lead, zinc, chromium, cadmium, and others as may be indicated by the specifications of the items being treated. This work will be performed by USAEHA as part of their activity at HWAAP. It is a significant factor in securing modifications to the WADF/HWAAP atmospheric emissions permit for the unit.

One main consideration for establishing the sequence shown was to avoid testing the same explosive twice in succession. That allows time for off-site analysis of process samples and for evaluation of the results in the event decontamination is not achieved and conditions must be modified. Another consideration is that the same item not be tested twice in succession (except Tests #4 and #5, #9 and #10, and #14 and #15) even where the explosive may differ, e.g., the 3" and 5" shells which are to be processed for decontamination of TNT, HBX, and Yellow D. Then, only a minimum number of racks has to be provided, with time available for them to be unloaded and refilled for a later test. A third consideration affecting the sequence shown was for trials with Yellow D (Ammonium Picrate) to begin later in the program. Yellow D is considered likely to be somewhat more hazardous and less predictable to process than TNT, RDX, and compositions based on them. The opportunity for gaining operational experience with the systems is considered desirable before Yellow D is decontaminated.

The first (and larger) group of HGD tests (Appendix D, Tests #1 through #20) will be performed with munitions items which have never been contaminated or have been previously decontaminated by conventional means and then recontaminated or "spiked" with the appropriate explosives, except for Test #16 and #19. The exact quantity of explosive material to be spiked in an individual item will be determined in baseline experiments as specified in Appendix B, Methods and Procedures. That will provide for determination of the proportionate residual level of contamination in the event that complete decontamination is not achieved in a test. However, it cannot be certain that spiking by deposition from solution will effectively simulate residual explosive contamination in a munition which was initially loaded by melting or pressing the explosive, followed by aging in inventory, followed eventually by removal of the bulk explosive charge by steaming, washing, or autoclaving. Therefore, tests will be concluded by processing demilitarized munitions where such munitions exist. This will confirm

the efficacy of the HGD process on the munitions to be processed in a future operational mode for the facility.

The test sequence emphasizes confirmation and confidence in decontamination efficacy as far as possible for the number of tests planned. If a test does not provide decontamination of the items processed, the next and subsequent tests with that item will be at more severe conditions of temperature and/or retention time. No effort will be made to optimize treatment conditions to conserve energy and reduce time; except, provisionally, for Yellow D. It is considered that such efforts would reduce the confidence level of decontamination efficacy by reducing the number of test replicates at conditions of initial or greater severity. The potential savings for the HGD facility as configured at WADF do not appear great in any event. The chamber is heated by recirculated oxidizer flue gas, with tempering air added at all times. Therefore the only energy cost for a higher operating temperature, within the system's capability, is the fuel consumed in the incremental time required to reach a temperature above the previous value. The operational approach currently envisioned for HGD production treatment is for day shift handling of munitions, with automated, unattended overnight heatup, steady state treatment, and cooldown of the chamber. Minor changes in treatment time would not affect such a schedule. Only large reductions in cycle time would be of value, and even they would require significant changes in plant personnel scheduling, calling for round the clock staffing to take advantage of them. Establishing the safe minimum values for treatment temperature and time would also likely require many more tests than planned.

### **3.3 Test Activity Responsibilities**

TVA will maintain technical direction and control of all field activities to be conducted during the HGD Test Program. The responsibilities of TVA and DZB to perform the various activities of this test program are planned so as to take advantage of each organization's capabilities. Assistance will be requested from other agencies and organizations as required for the conduct of the program. Safety, efficiency, and quality of test procedures and results are considered essential.

DZB will have operating responsibility for the HGD facility. They will set aside designated items from material at hand, spike test items by the TVA procedure, handle all test items for loading, unloading, and for any sampling procedure calling for hoist or other handling equipment to give access to the item for sampling. DZB will stockpile the test items prior to testing and after treatment in case

additional examination is called for. DZB will decontaminate the test items and release them for disposal. DZB will operate the HGD chamber, its controls, and all auxiliary equipment. DZB staff will maintain all operating and control equipment. Assignment of a maximum of test activities to DZB personnel will minimize costs by employing site personnel, and increase program safety and efficiency by virtue of their site-specific training and experience.

DZB will be responsible for providing TVA with system operating data at the conclusion of each test run. Data will include, as a minimum, date, time, test number, temperature profile of each thermocouple, duration of test, and description of problems encountered and recommendations for corrections.

TVA field personnel will take all test samples, whether from items about to be processed (in order to confirm the presence of spiked or residual explosive), processed items, HGD facility surfaces, and gas streams from the chamber to the oxidizer, from the oxidizer stack, and from the hot gas recirculation system. DZB personnel will assist only by lifting, moving, and handling items for access and agitation. TVA will identify samples and ship them to the TVA analytical laboratory. TVA field crew will perform QC measures agreed upon by TVA and USAEC such as inclusion of blank and unknown samples of solvent and record keeping. TVA will monitor HGD system operating parameters and retain copies of raw data pertaining to treatment conditions and equipment operating parameters, and ensure that correspondence between operational data and process samples is maintained. TVA field crew will maintain records of data from the installed Continuous Emissions Monitors (CEM's) and TVA's field crew will maintain a daily log of all activities pertaining to each test.

TVA laboratory personnel will perform chemical analysis of the process and QC samples in accordance with previously established and agreed on methods (see Section 3.4.5 and Appendix B for Methods and Procedures). TVA will perform laboratory procedures as established with USAEC, including, but not limited to, analytical instrument maintenance and calibration, and spiking of samples with a surrogate compound to demonstrate instrument performance on each individual sample. TVA laboratory personnel will evaluate results for analytical validity and statistical consistency. Results will be forwarded by telefax to the TVA field office where they will be employed for evaluation of the efficacy of decontamination in the particular test. They will apply those results to the conduct of future tests with the same explosive. As described above, the main consideration of such evaluation would be the requirement for more severe



treatment conditions in case of a failure to decontaminate a given item or explosive. A secondary, but still significant consideration of concurrent test evaluation will be the measures to be taken in the event of failure of various quality control samples to meet expected criteria. In the laboratory, failure of surrogate recovery on quality control spike samples (other than matrix spikes) would indicate the need for reanalysis for all analytes. Failure of a continuing calibration check would indicate the need for reanalysis of subsequent samples from the last satisfactory calibration. Failure of matrix spike or matrix spike duplicates indicates the need for a review of data, but not necessarily for reanalysis provided some potential for matrix interference is indicated. Presence of target compounds in blank samples would indicate the presence of contamination and the need for reanalysis unless there was indication that the contamination occurred in the field. In that event, field practice for taking and processing samples would be reviewed and corrected. Review of sampling activities and analysis of field supplies of solvents and standard solutions for contamination or deterioration may also be indicated. Continuing poor analytical quality from the laboratory on quality control spike samples would necessitate interruption of the test sequence until problems were resolved.

Other agencies and organizations have defined responsibilities for the HGD tests. USAEC will supply Standard Analytical Reference Materials (SARMS) of the explosives being tested, and will provide consultation on chemical and analytical considerations. USADCS provided explosives safety training, and will perform a review of test results as to safety and efficacy. HWAAP will provide samples of other explosives and non-explosive materials for methods development, and will interface between TVA and DZB for facility requirements. USAEHA will take stack gas samples for air quality considerations and for facility permitting as required for operational activity.

### **3.4 Test Procedures**

#### **3.4.1 Preparation of Items**

Selection. As discussed earlier, the test plan calls for two distinct types of tests: (1) spiking tests, in which the test items have been spiked with explosive and (2) tests in which the actual items that would require routine decontamination are used. Each of these two types of tests have different item requirements.

In all tests employing spiked items, only items that have been through the flash furnace (FF-13) will be used (unless noted otherwise), both for the spiked items themselves and for any inert items that are loaded into the test chamber. For an

exception see MK 25 ship mines. MK 25's have not been through FF-13. This will ensure that the total quantity of explosive that enters the chamber is known. After the spiking test series has been completed, various tests in the test plan will employ items that have been demilitarized thus simulating the actual HGD of items in production mode. (See Table 3 for sequence and explosive compound.)

For spiking and sampling purposes, the test items fall into two categories: (1) the projectile-type items, which have a small internal surface area and are enclosable, thus allowing a small volume of liquid solvent to be used to contact and extract explosives from the entire internal surface, and (2) large munitions-type items, which have too much internal surface area to allow liquid rinsing of the entire surface to be practical. In this latter case, specific areas within the items will be designated for spiking and sampling purposes. Six items and five explosives have been selected for HGD testing as shown in Table 1 in Section 1.4.1.

Spiking. The explosive concentration and the quantity of the spiking solution (per spike) will be predetermined during the baseline studies work (discussed earlier in Section 2). The concentration will be verified by analysis. The quantity of solution for each spike will be kept uniform by using identical pipettes that are each singularly marked at the proper volume.

It is desirable to spike test specimens with the explosive normally to be decontaminated from that item. That can be reasonably well accomplished with most of the explosives of interest--TNT, Comp B, HBX, and Yellow D. It has not proven practical for Comp A-3, however. That explosive comprises 91 percent RDX and 9 percent of a wax stabilizer. No solvent or miscible combination of solvents has been identified which will produce a homogeneous solution of Comp A-3. Therefore, spike tests with items normally loaded with Comp A-3 will be spiked with neat RDX. The substitution has been evaluated and approved by USAEC.

DZB will prepare the spiking solution and carry out the spiking in accordance with the procedure developed by TVA and shown in Appendix E.

Confirmation of spike/contamination. For each loading of the HGD test chamber, one spiked projectile or spiked area will be randomly selected for spike confirmation (i.e., assurance that the spiking was properly performed). For the projectile tests, the selected projectile will be held out of the chamber for later sampling and analysis. (A suitable inert item will be used to replace the spiked

item in the chamber.) For the tests employing spiked areas, the selected area will be sampled with solvent-laden wipes, and the wipes will be subsequently analyzed as outlined in Appendix B. In both cases, the random selections will not be known in advance by the personnel who perform the spikings.

#### 3.4.2 **Positioning and Lading of Railcar**

Typical positioning and lading diagrams for the items to be tested are shown in Table 4. All figures are included in Appendix D.

**TABLE 4**  
**TYPICAL RAILCAR POSITIONING/LADING DIAGRAMS**

<u>Test Item</u>	<u>Figure #</u>
Railcar Placement Inside Chamber	D-1
Typical Pallet Arrangement on Railcar	D-2
3" Projectile Racks Arranged on Pallet	D-3
5" Projectile Racks Arranged on Pallet	D-4
106mm Projectile Racks Arranged on Pallet	D-5
175mm Projectile Rack Arranged on Pallet	D-6
MK 54 Depth Bombs (Sawed Ends) Arranged on Pallet	D-7
MK 25 Ship Mines Arranged on Pallets	D-8

The following discussion illustrates a typical railcar lading procedure using the 175mm spiked projectile as an example from Figures D-6 and D-13. Twenty-four spiked 175mm projectiles will be delivered to a staging area for lading. For spike confirmation, one of the projectiles will be sampled prior to being loaded on the railcar. This random sample will identify the explosive compound going into the chamber. Once the projectile is sampled it will be loaded onto the railcar for treatment. That particular item will be resampled after decontamination to verify the process. (Sampling procedures are discussed in Section 3.4.4.)

The remaining 23 projectiles will also be positioned on the railcar according to a predetermined layout plan as shown in Appendix D (see D-13). A full railcar load of 175mm projectiles calls for 8 racks, each containing 12 projectiles, with the racks laid out in a 2 by 4 rectangular grid arrangement. All racks will be placed on heavy wire pallets to prevent the test items from coming in contact with the railcar bed. The 24 projectiles to be sampled will be divided such that all of the

racks will receive three projectiles each. The positioning of the projectiles in the 12 slots within a rack was determined randomly and illustrated in Figures 9 and 10. (A plan was considered where the 24 positions would be selected randomly from the 96 available slots at large, but it was rejected since it would be unacceptable if the random selection process put a disproportionate share of the projectiles at one end of the railcar.)

As the spiked projectiles are loaded on the railcar they will be marked (using a high temperature marker) with the test sequence number and a unique identification number which can be cross-referenced to the projectiles' positions. After the spiked projectiles have been loaded, the remaining projectile slots are filled with inert projectiles to provide thermal mass, thus ensuring the presence of temperature distribution patterns across the railcar. Once the railcar is positioned in the chamber, at least one thermocouple will be attached to the inner surface of each spiked projectile. The output from each of these thermocouples will be recorded throughout the test, ensuring that there will be a separate "thermal history" of each spiked projectile after the test run is complete.

The same general procedure will be employed for 3" projectiles, 5" projectiles, and 106mm projectiles (Figures D-3, D-4, and D-5 respectively) which are the test items for TNT, RDS, HBX, Yellow D, and Comp A-3. As shown in Figure D-12, the two calibers will be tested together, with racks of the appropriate size alternating on the railcar. A test will employ 24 spiked projectiles, plus confirmation samples. An additional 168 inert projectiles will comprise the car lading. The approach for random placement will be followed as shown in Figures 7, 8, 11, 12, and 13.

Large munitions items such as the MK 54 depth bombs (sawed ends) and MK 25 ship mines (cut in half) will be placed on racks on the railcar as shown in Figures D-7 and D-8, respectively. Due to the smaller number of these larger items that will comprise a carload, all pieces will be contaminated either by spiking or as they come from the WADF. All pieces will be treated and wipe sampled. A wipe sample will be taken off one large item at random to confirm spiking (or contamination in the tests with actual contaminated items.) Thermocouples will be placed at selected points on and around the items to provide for a thermal history of that particular lading and individual test. The location of each thermocouple will be recorded on each individual test diagram.

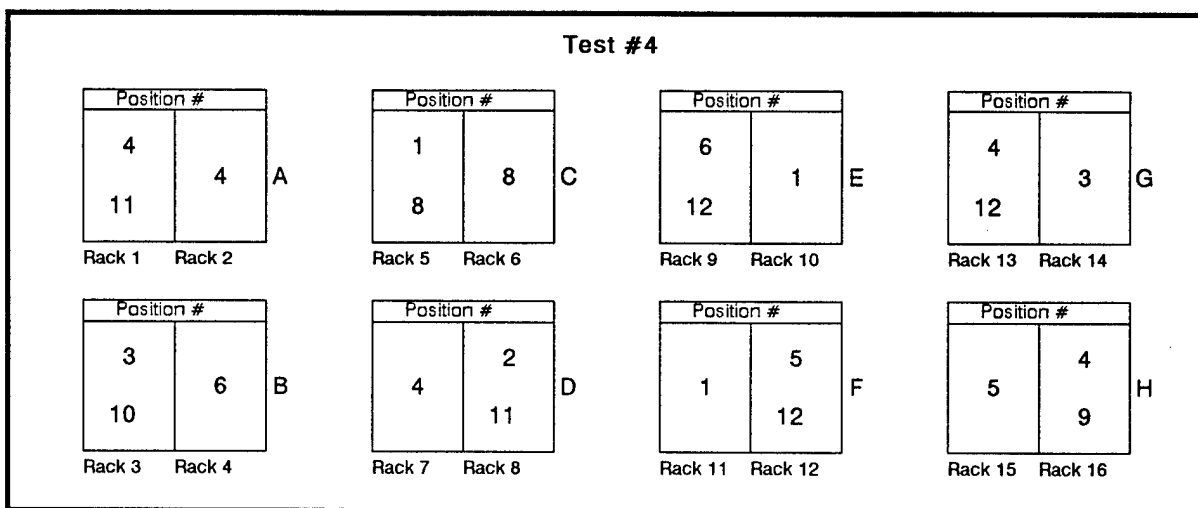
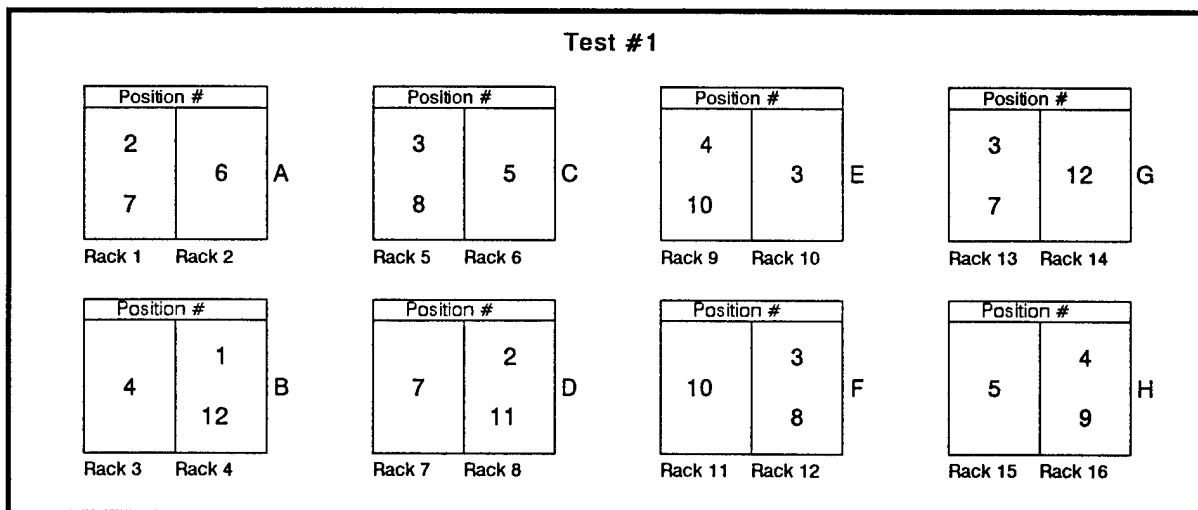


Figure 7 – Random Sample Locations for 3" Projectiles – Spiked with TNT

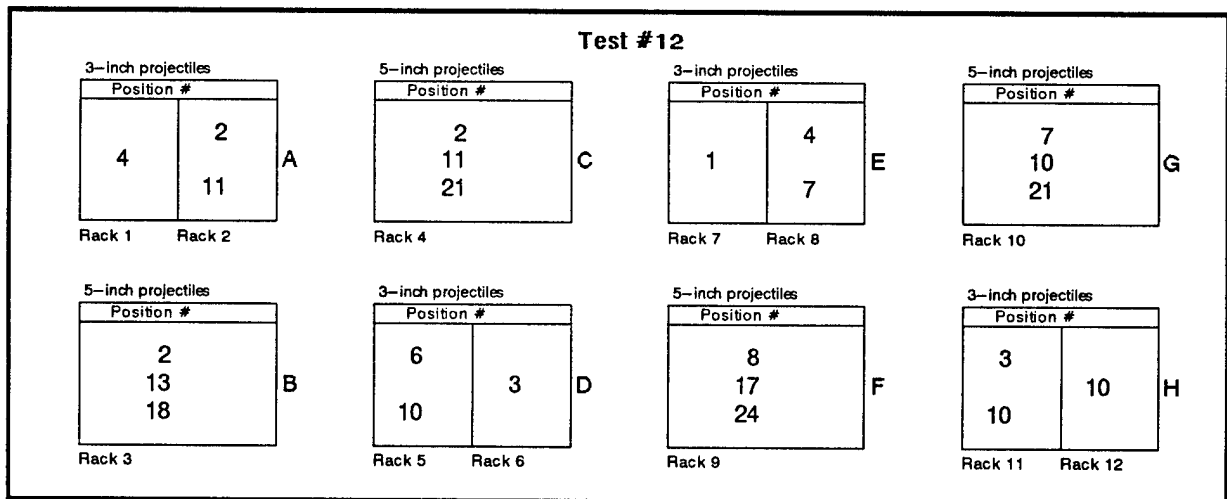
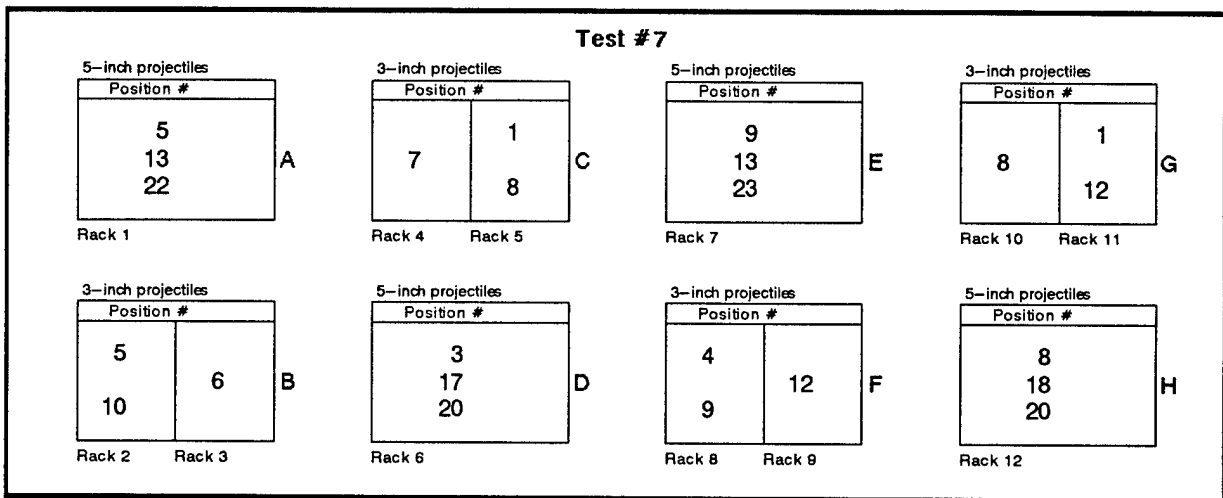
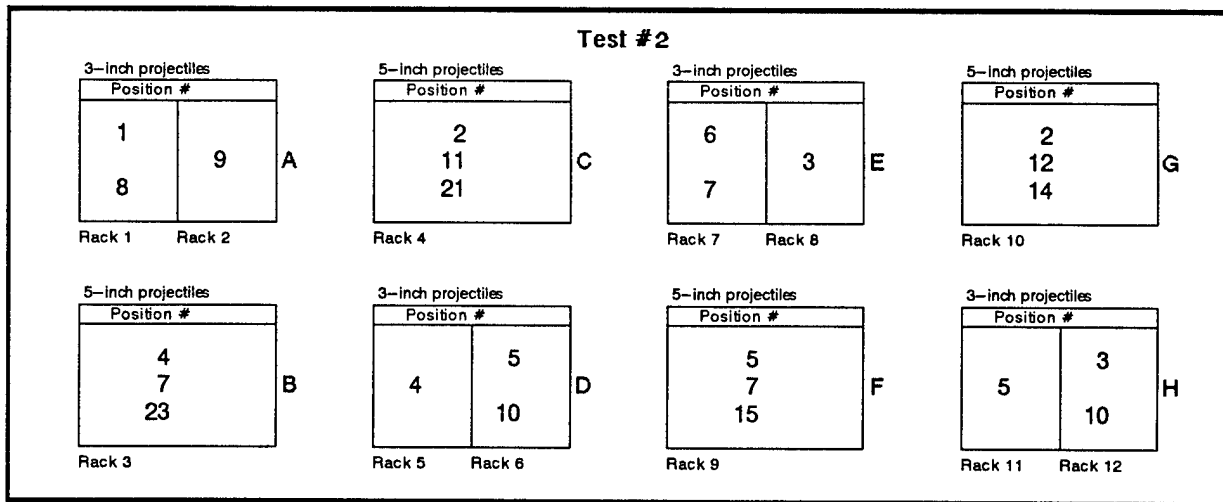


Figure 8 – Random Sample Locations for 3"/5" Projectiles – Spiked with RDX

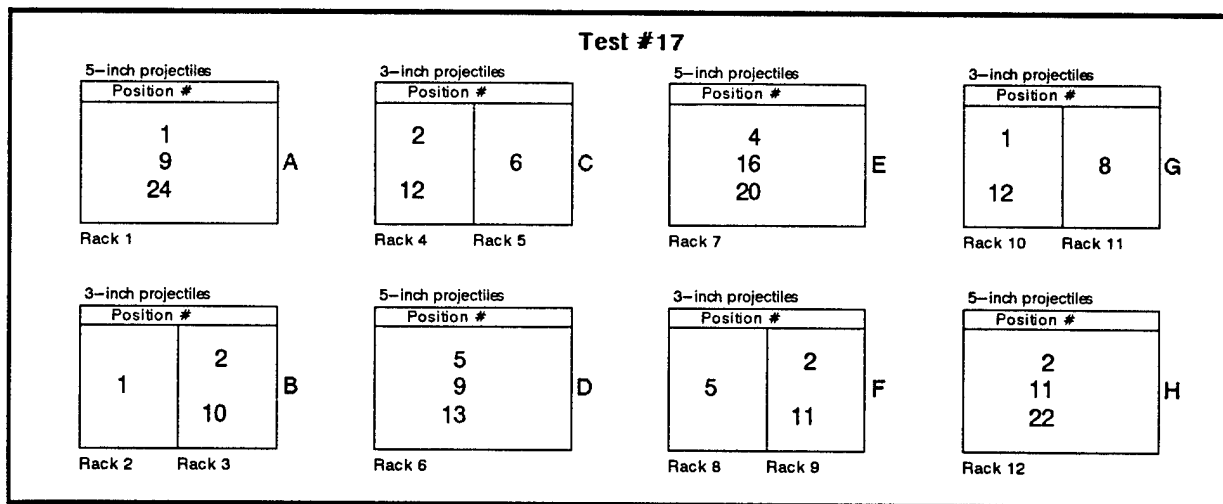


Figure 8 (Continued) – Random Sample Locations for 3"/5" Projectiles – Spiked with RDX

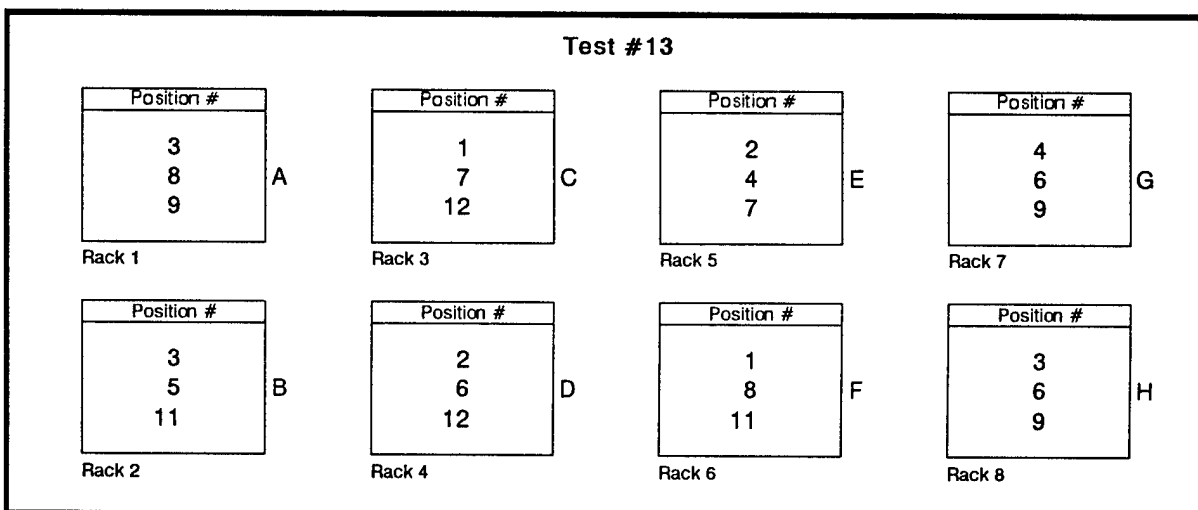
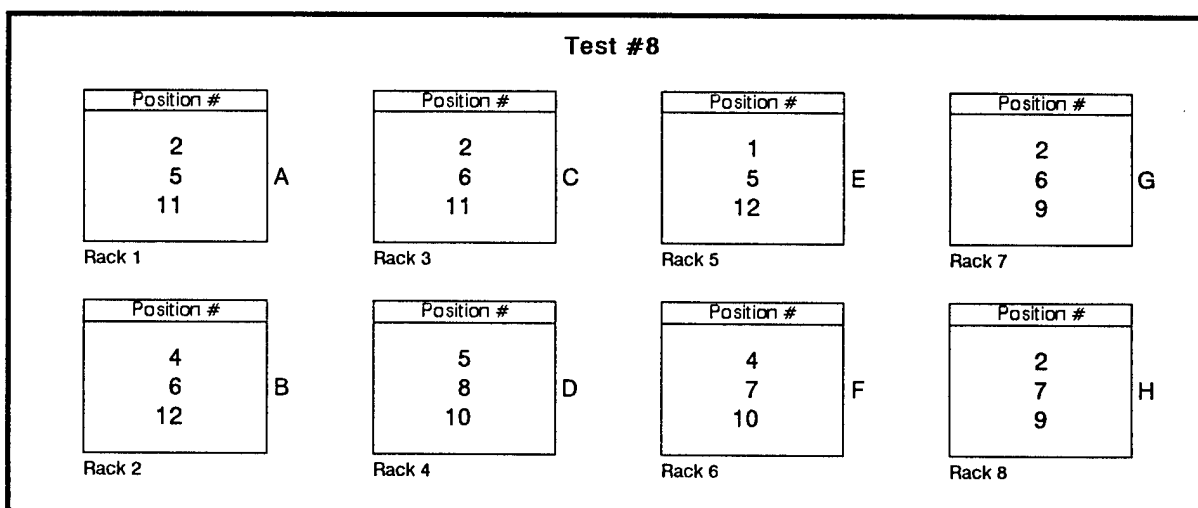
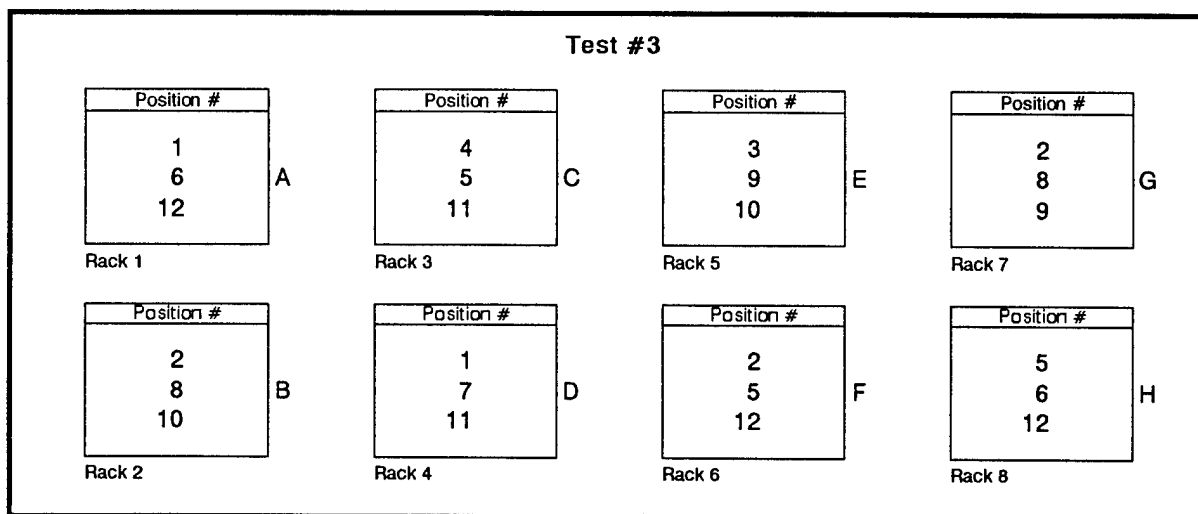


Figure 9 – Random Sample Locations for 175mm Projectiles – Spiked with Comp B



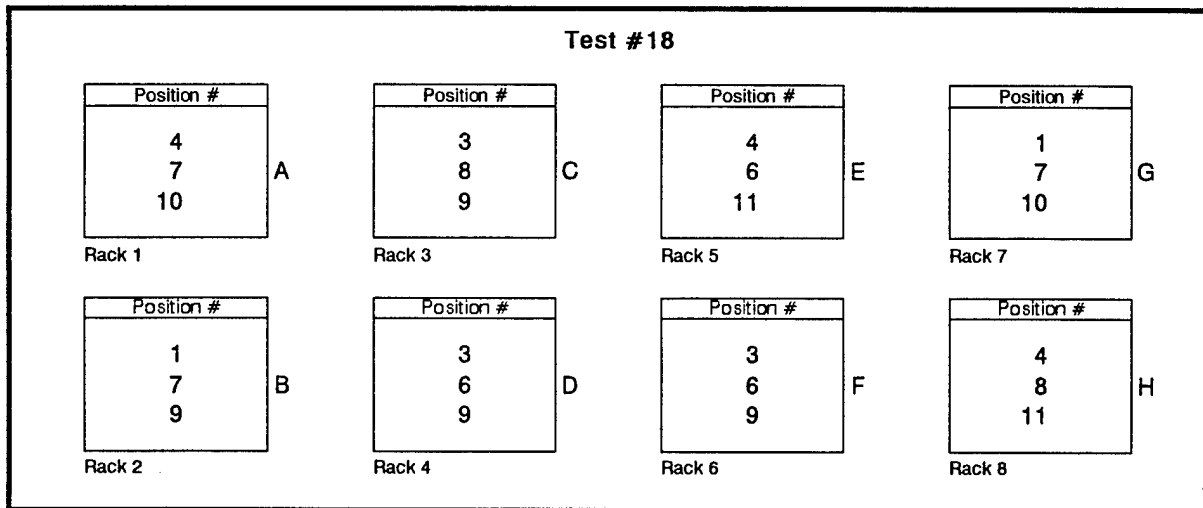


Figure 9 (Continued) – Random Sample Locations for 175mm Projectiles – Spiked with Comp B

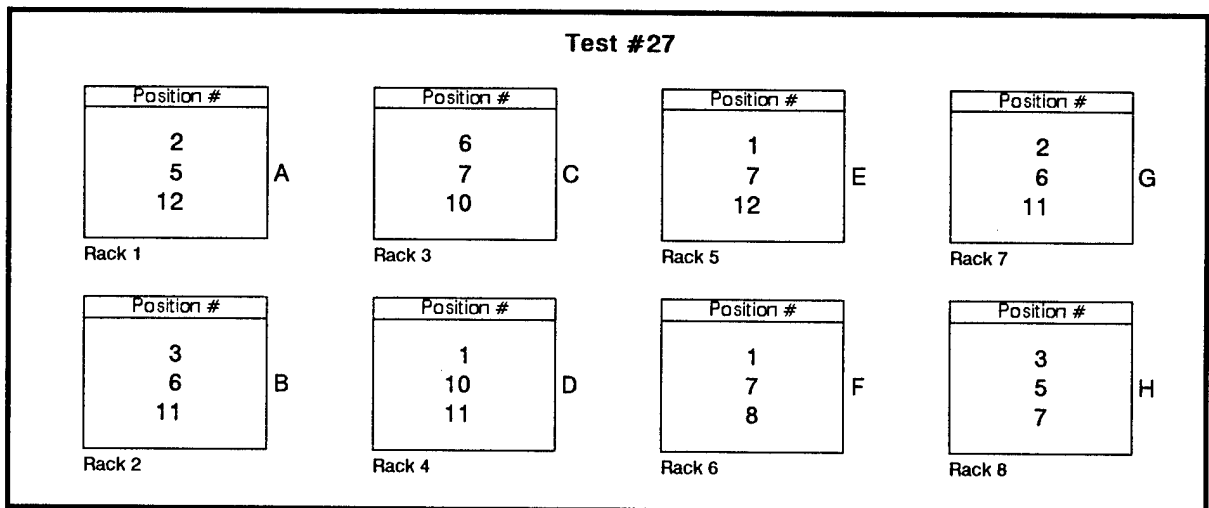
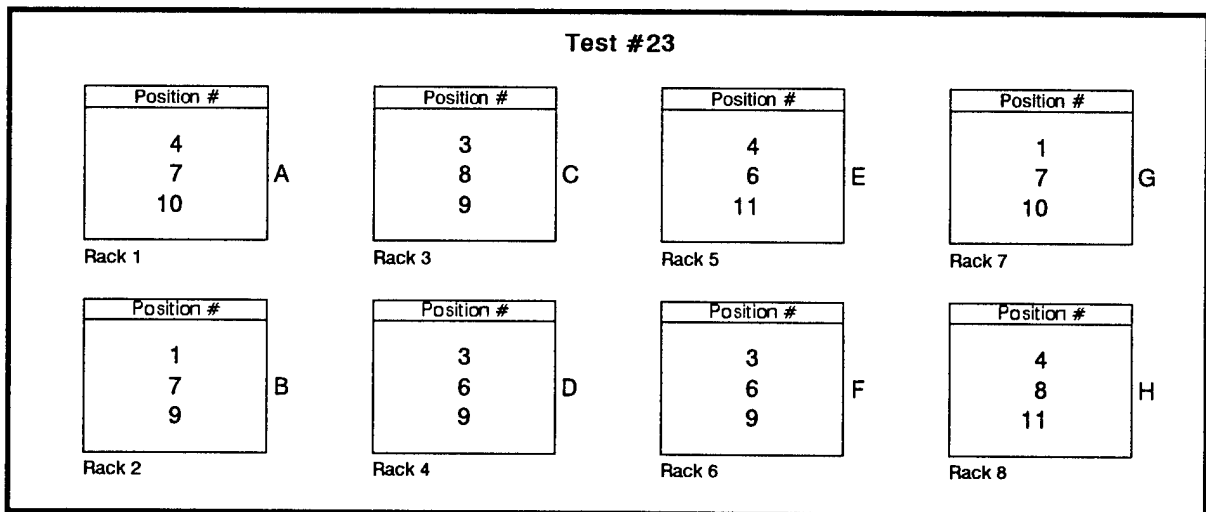


Figure 10 – Random Sample Locations for 175mm Projectiles – From Demil with Comp B Residue

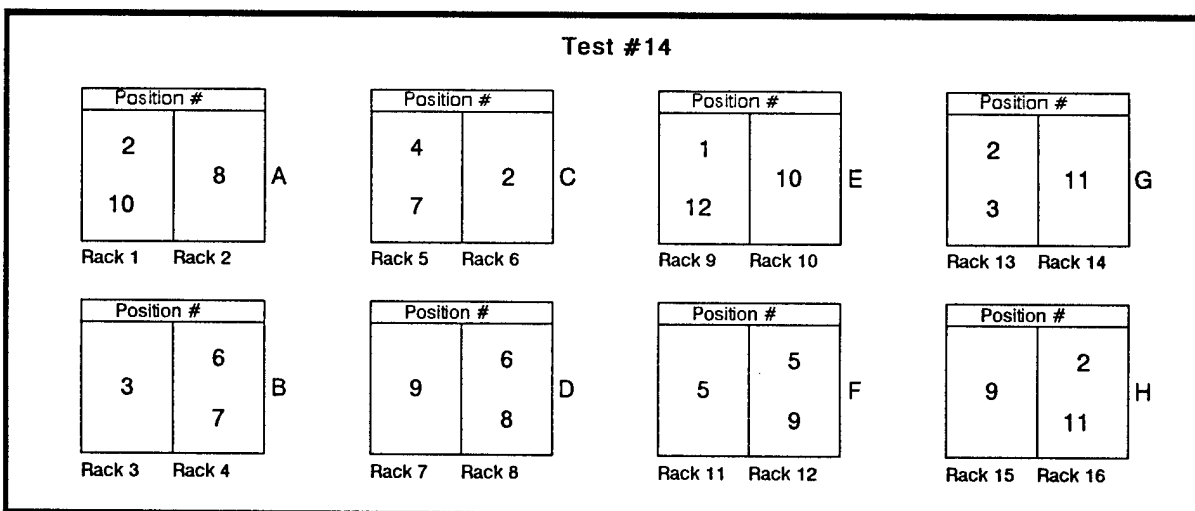
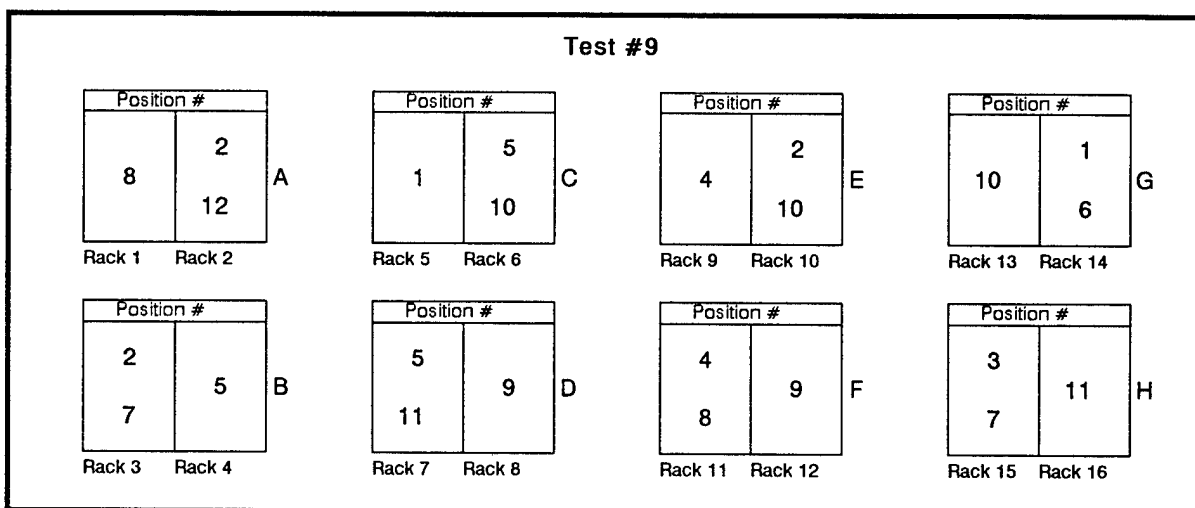
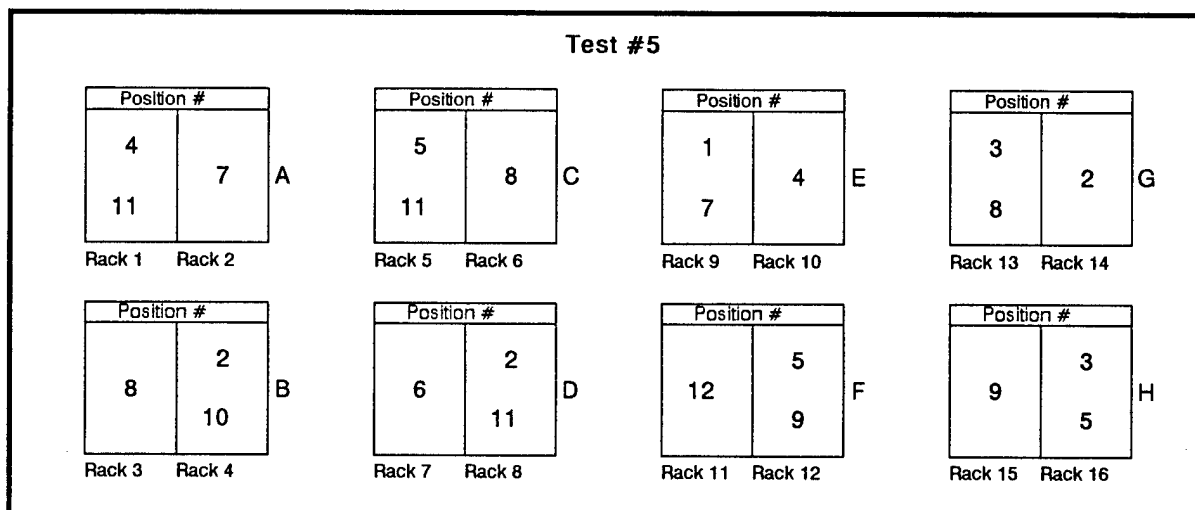


Figure 11 – Random Sample Locations for 3" Projectiles – Spiked with HBX  
HGD of Explosives Test Plan 3-18 Hawthorne AAP

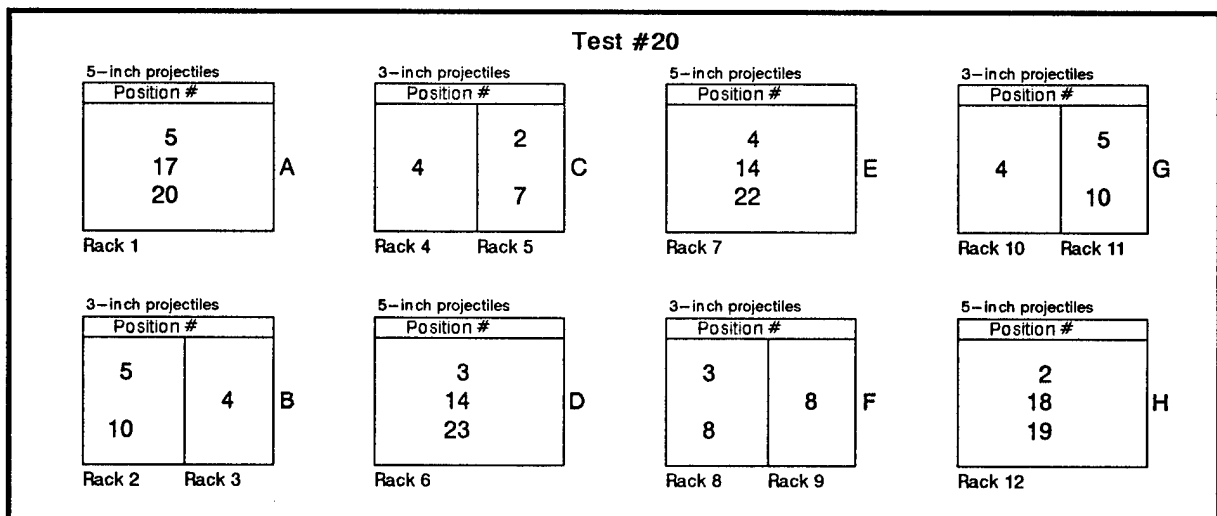
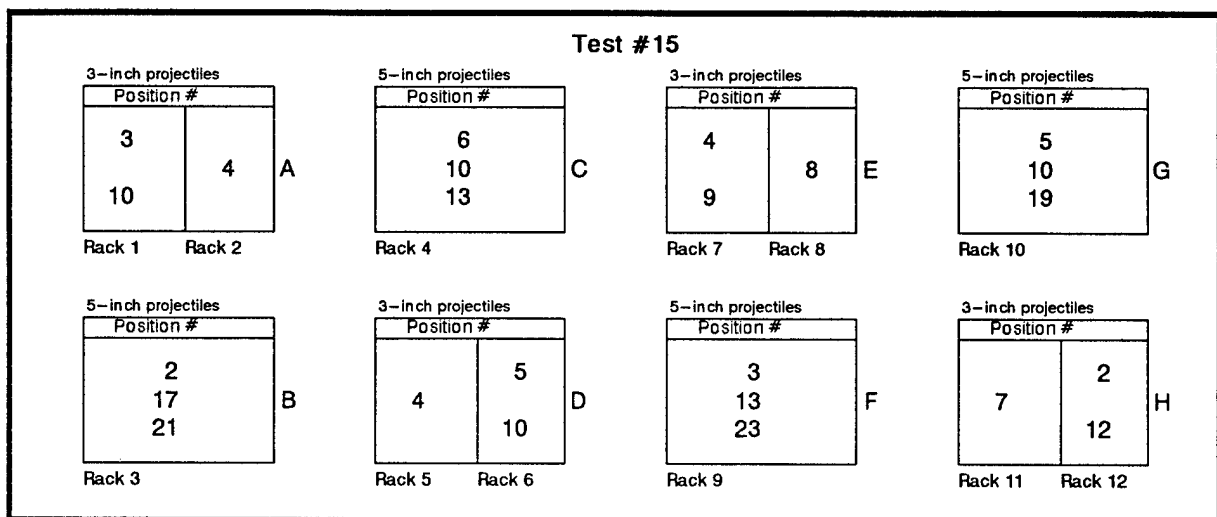
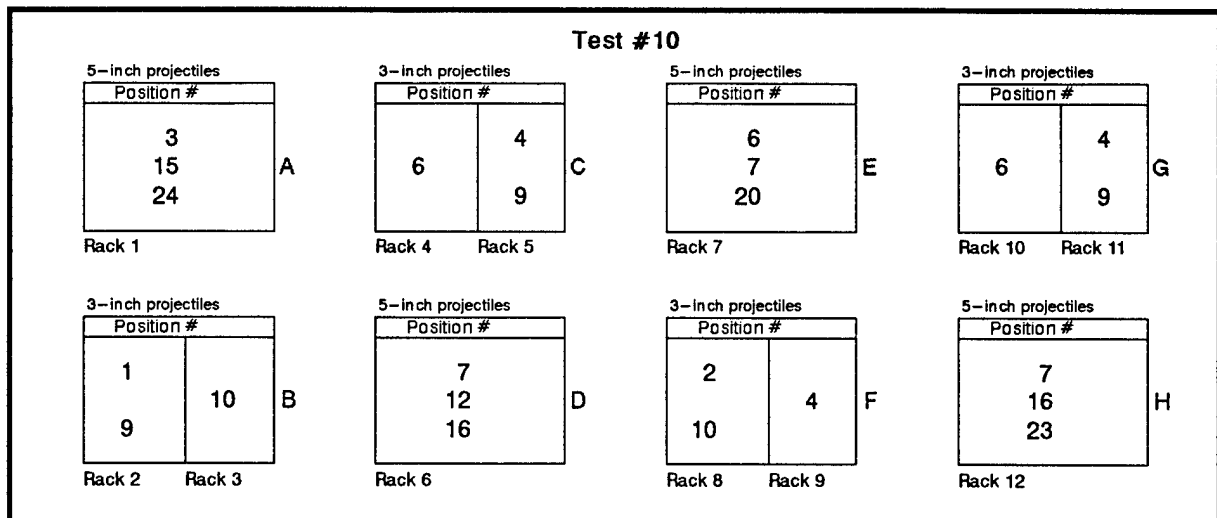


Figure 12 – Random Sample Locations for 3"/5" Projectiles – Spiked with Yellow D

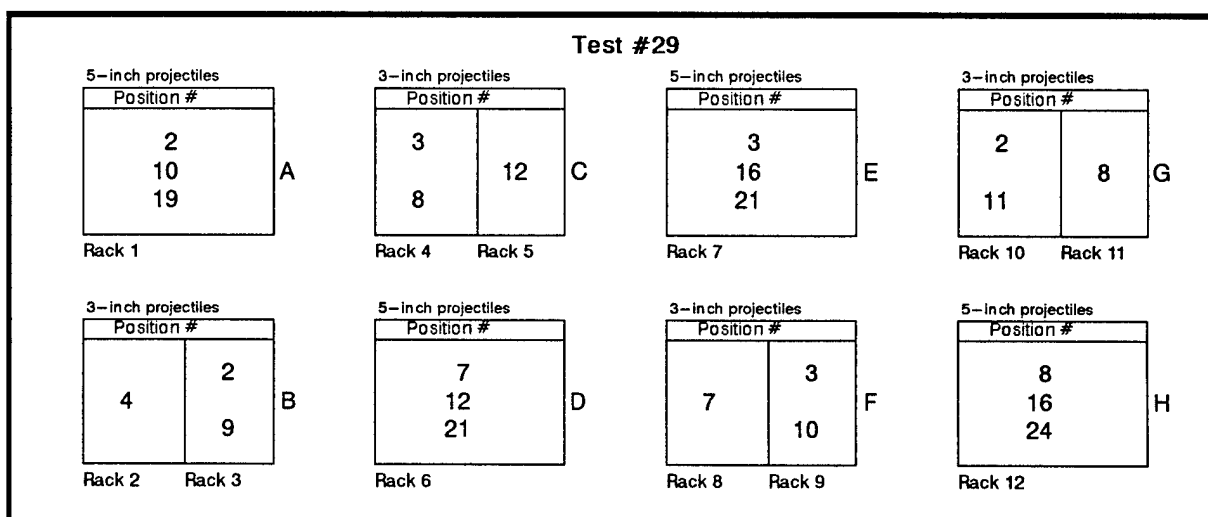
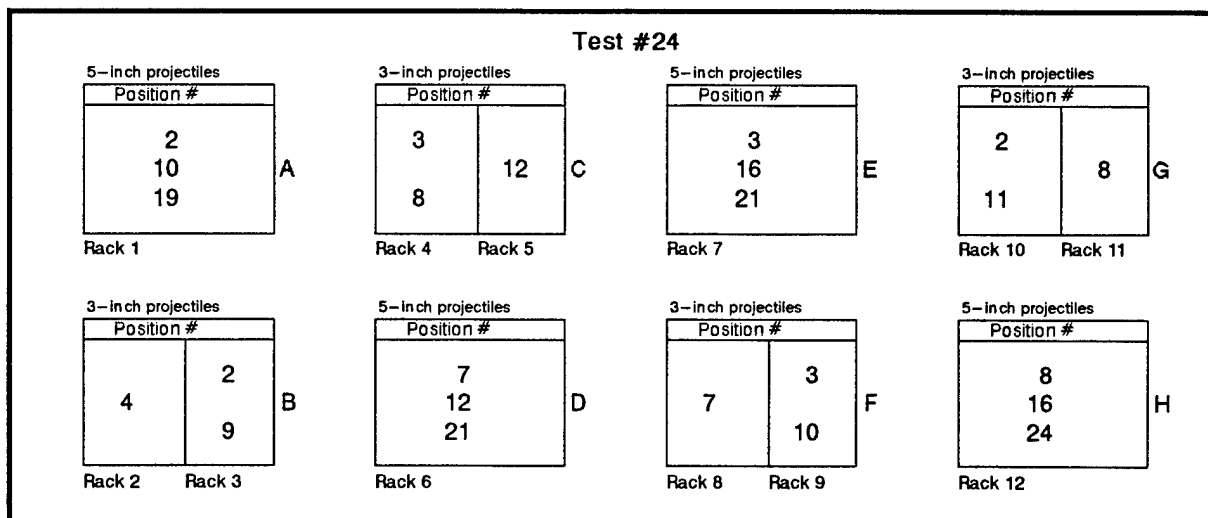


Figure 12 (Continued) – Random Sample Locations for 3"/5" Projectiles – Spiked with Yellow D

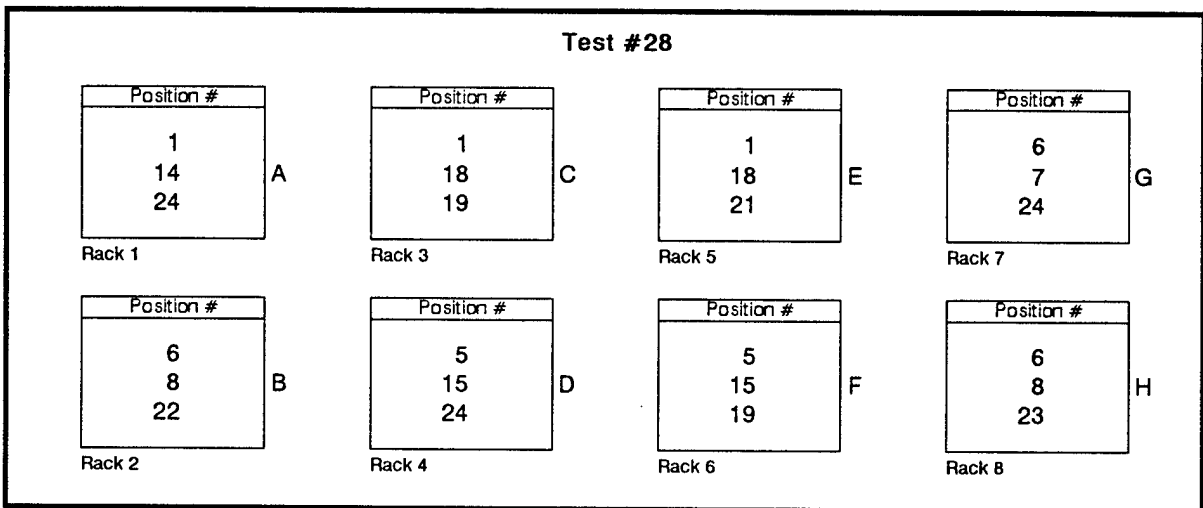
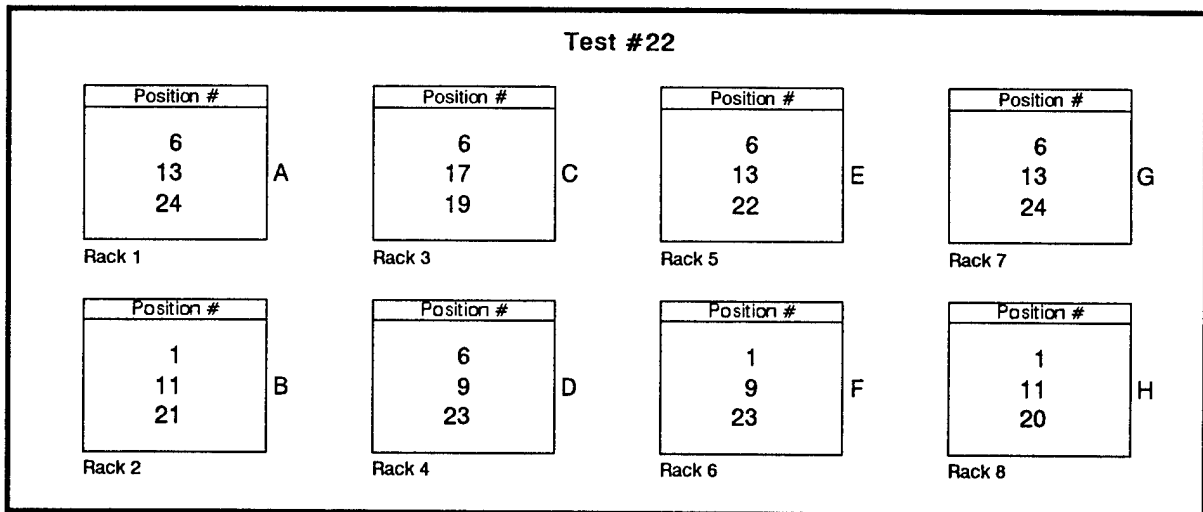


Figure 13 – Random Sample Locations for 106mm Projectiles – From Demil with Comp A-3 Residue

### **3.4.3**

#### **Operation of the HGD System**

The railcar will be moved into the HGD chamber and thermocouples fitted to spiked items and to other locations inside the chamber. All thermocouple locations will be recorded on the appropriate test diagram. The door will be closed, a safety inspection of the chamber and surrounding area will be conducted, and the Maples controller program will be initiated to start the test. It will automatically start the system burner, blowers, and controllers to bring the chamber up to the planned operating temperature, hold the chamber at operating temperature for the scheduled treatment time, and then cool the chamber and its contents by circulating ambient air.

When the chamber has cooled sufficiently to allow opening the door, the railcar will be moved out to allow the test items to air cool. Ambient air will continue to be blown through the chamber to cool the structure to a temperature low enough to permit personnel entry and conduct surface sampling. Samples will be taken as described below. A railcar with the next lot of test items will be positioned for treatment. The next test will start as soon as possible but time will be allowed for collection of all item, surface, and gas samples, collection of raw data from Maples controller, CEM and temperature monitors, and an examination of the facility for maintenance requirements.

### **3.4.4**

#### **Sample Protocol**

#### **3.4.4.1**

##### **Sampling of Items**

Items are sampled immediately after they have been removed from the chamber and allowed to cool. The sampling procedures are the same for both spiked projectiles and demilitarized projectiles. However, for the large munition items, the sampling of some of the items will require special procedures to be used because of the presence of an asphalt coating on the item interior.

In the case of a projectile, a measured weight of extraction solvent (acetonitrile for TNT and RDX; distilled water for ammonium picrate) is poured into the projectile. The volume must be sufficient to wet and rinse the entire internal surface and leave a volume for analysis, but must also be as small as is practicable to maximize the explosive concentration for analysis accuracy. (The baseline study work will provide guidance as to the appropriate volume.) The projectile is plugged or capped, and then tipped and rolled to promote thorough rinsing of the internal surface. The solvent is then poured (or pipetted) from the projectile into a precleaned sample jar. Further extractions to minimize this explosive retention are not considered desirable since they will be very dilute and

thus susceptible to analytical inaccuracy. It will be advantageous to remove as much solvent (and therefore explosive) on the first extraction.

However, all sampled items will be temporarily set aside and stored in case further extractions are desired.

For those items where a specific area is sampled, gauze wipes saturated with solvent will be applied directly to the area to make the extraction. (Again, the baseline study work will provide guidance as to the specific procedure, solvent, and number of wipes to be used. EPA wipe sampling technique will also be applicable and will be followed.) To produce a liquid volume for analysis, the sample wipes will be rinsed with fresh solvent under suction.

In addition to the samples from the items, each test run will include quality control samples introduced by TVA field personnel. These will include solvent blanks, unknown quality control samples at two concentrations (nominally 2 and 50 parts per million), and the spiking solution for verification of concentration. Field duplicates may be introduced where possible. One blank and one each of the two unknown quality control samples will be introduced for each set of twenty samples. A sample of the spiking solution will be retained for each run. It will be analyzed, if necessary, to resolve a discrepancy.

Using the example of a test with 175mm projectiles, a full set of test samples will include the following:

- 24 regular test samples
  - 1 spiking solution for retention
  - 2 blank samples
  - 2 nominal 2 parts per million samples
  - 2 nominal 50 parts per million samples

In this example, since more than twenty samples were taken, two sets of field quality control samples were introduced.

Logs and records shall be kept of the weights and volumes of material and solvent used in mixing the two unknown quality control samples. Actual concentrations may be adjusted to other than 2 and 50 parts per million as required by the needs of the process.



Measures shall be taken to ensure sample numbers are randomly assigned so that the laboratory will not know the actual sample concentration beforehand nor shall it know which samples are quality control samples and which are test samples.

Table 5 is a tabulation of all tests indicating the number of items required and the number and type sample required for that test. Tables 6a, 6b, and 6c are summaries of test items and type samples required.

#### **3.4.4.2 Samples from Facility Surfaces**

Samples will be taken from selected facility surfaces to ascertain whether or not there are cold spots in the facility which are capable of condensing explosives out of the hot gas stream. These cold spots could present a hazard to personnel or the facility, since, during routine production, the explosives would tend to collect and build in these places.

Seven locations have been identified as having a potential for cold spots. These are:

- The chamber floor.
- The chamber walls.
- The entrance to the hot gas outlet duct.
- The hot gas outlet duct elbows.
- The fan blades in the induced draft (ID) fan.
- Cold spots on items identified.
- Rail adjacent to door.

During the course of Test Run B, temperatures will be monitored at various locations in an attempt to identify cold spots. All of these locations will be recorded for each test run and sampled in specified areas using solvent-laden wipes after each test run. The floor, walls, and duct entrance are all accessible from inside the chamber. The duct elbow will be accessed from a specially installed 4-inch nozzle that will allow access to obtain a wipe from the internal elbow surface. The ID fan will require removal of an access plate on the fan housing to obtain wipe samples from the blades or internal surfaces. See Tables 5 and 6 for quantities of samples to be taken.

Background samples will be taken before the first of this series of tests with actual explosives in order to determine whether exploratory operation of the system may have already left deposits of explosives or other contaminants on chamber surfaces.

TABLE 5  
ITEMS AND SAMPLES REQUIRED

Test No	Test Item	Explosive Compound	Items req'd for each test			Number of Samples to be taken										Total																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																													
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TABLE 6a  
SUMMARY - ITEMS AND SAMPLES REQUIRED - NO EXPLOSIVES

Test No	Test Item	Explosive Compound	Items req'd for testing					Number of Samples to be taken															Total Samples																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																										
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TABLE 6b  
SUMMARY - ITEMS AND SAMPLES REQUIRED - SPIKED ITEMS

Test No	Test Item	Explosive Compound	Items req'd for testing				Number of Samples to be taken														Total Samples				
			Spiked	Demil	Inert	Total Items	Air Samples		Extract (Solution) Samples			Wipe Samples													
							Duct	Stack	Conf.	Blank	Unk 2	Unk 50	Items	Conf.	Blank	Unk 2	Unk 50	Items	Duct	Elbow		Fan	Floor	Chamb	Walls
1.4	3" Projectiles	TNT	48		168	216	4	4	2	4	4	4	48					2	2	2	2	2	2	2	84
2.7, 12, 17	3" Projectiles	RDX	48		84	132	8	8	4	8	8	8	48					4	4	4	4	4	4	4	120
	5" Projectiles	RDX	48		84	132							48												48
3.8, 13, 18	175mm Projectiles	Comp B	96		72	168	8	8	4	8	8	8	96					4	4	4	4	4	4	4	168
5.9, 14	3" Projectiles	HBX	72		168	240	6	6	3	6	6	6	72					3	3	3	3	3	3	3	126
6.11	MK 25 Ship Mines (Half)	TNT/not melt	16		16	16	4	4										2	2	2	2	2	2	2	78
10, 15, 20, 24, 29	3" Projectiles	Yellow D	48		84	132	10	10	5	10	10	10	60					5	5	5	5	5	5	5	150
	5" Projectiles	Yellow D	48		84	132							60												60
26, 30	MK 25 Ship Mines (Half)	Comp B		16		16	4	4						2	4	4	4	4	48	2	2	2	2	2	84
	Totals						44	44	18	36	36	36	432	4	6	6	6	6	96	22	22	22	22	22	918

TABLE 6c  
SUMMARY - ITEMS AND SAMPLES REQUIRED - ITEMS FROM DEMIL

Test No	Test Item	Explosive Compound	Items req'd for testing				Number of Samples to be taken																Total Samples		
			Spiked	Demil	Inert	Total Items	Air Samples		Extract (Solution) Samples				Wipe Samples												
							Duct	Stack	Conf.	Blank	Unk 2	Unk 50	Items	Conf.	Blank	Unk 2	Unk 50	Items	Duct	Elbow	Fan	Floor		Chamb	Walls
16, 19, 21, 25	MK 54 Depth Bombs (Ends)	HBX		96		96	8	8					4	8	8	8	8	96	4	4	4	4	4	4	168
23, 27	175mm Projectiles	Comp B		48	72	120	4	4	2	4	4	4	48					2	2	2	2	2	2	2	84
22, 28	106mm Projectiles	Comp A-3		48	168	216	4	4	2	4	4	4	48					2	2	2	2	2	2	2	84
31	HWAAP/DZB						4	2	1	4	4	4	30					1	1	1	4	4	2	2	64
	Totals						20	18	5	12	12	12	126	4	8	8	8	96	9	9	9	12	10	10	400

(1) Samples for test #31 are estimated - actual number of samples will be determined by number of tests conducted and number of items in chamber.

Investigation will be made to develop a simple spot test utilizing the color reaction of ethylenediamine with the target compounds. In this case, smears may be taken and examined on site to test suspected cold spots or unexpected deposits in addition to the smear samples sent to the laboratory for analysis.

#### **3.4.4.3 Gas Samples**

Gas samples will be taken during each test after effective decontamination conditions have been established for a given explosive. At scheduled intervals during testing, gas samples will be taken by a technique based on EPA Modified Method 5. Sampling will be applied to the discharge duct from the HGD chamber. Samples of absorptive resins and impinger solutions will be analyzed for explosives and related compounds. Exhaust duct samples in combination with stack samples taken by AEHA will provide for determination of the Destruction and Removal Efficiency (DRE) of the process.

A continuous emissions monitor (CEM) will be installed at a sample point on the chamber discharge duct. It will provide a continuous, time-keyed record of inorganic combustion products such as CO<sub>2</sub>, CO, and NO<sub>x</sub>. The CEM data will show the effective composition of the chamber atmosphere, indicating the accumulation of flue gas by continued recycling. It may also give an indication of oxidation of explosive compounds at the relatively mild conditions of the chamber interior.

The United States Army Environmental and Health Agency (USAEHA) has been requested to provide stack gas sampling for environmental considerations. USAEHA's sampling and analysis data will be used to modify the existing facility air permit before the HGD system becomes operational for full scale production. Emissions of CO, NO<sub>x</sub>, SO<sub>x</sub>, particulates (total and under 10 micron), and lead will be measured. USAEHA will sample the stack during the late stages of the HGD test program when demilitarized items are being decontaminated. This will allow time for reliable operating procedures to be established in the earlier parts of the program. It will also provide for sampling to take place while items actually contaminated with explosives residues are being processed, rather than the spiked items used for early tests under artificial control of the level and purity of contamination.

### **3.4.5      Analysis Protocol**

#### **3.4.5.1    Shipment**

All of the samples taken from one test will be marked, identified, and logged as described above and as further described in Appendix A. They will be shipped together, in approved sample coolers, by air to the TVA laboratory for preparation and analysis.

#### **3.4.5.2    Records**

Records will be kept in accordance with appropriate chain of custody procedures, based on guidelines shown in Appendix B.

#### **3.4.5.3    Analytical Procedures**

Samples will be analyzed by Method 8330 or the Picrate analysis procedure, as revised during the baseline studies and shown in Appendix B, depending on the explosive being tested.

#### **3.4.5.4    In-Progress Reporting**

Verified analytical results will be reported to the TVA field personnel by telefax in order to facilitate concurrent review and revision of the test program. This is needed in order to provide the maximum of confirmation and confidence in the efficacy of the process, based on the limited number of full facility tests with any one item and explosive.

### **3.4.6      Review and Evaluation**

The analytical results from a particular test will be correlated with sample sources. Analyses of blank and laboratory unknown samples will be confirmed for QC purposes. Analyses of process samples taken from munitions items being treated will be read to evaluate the efficacy of the decontamination process at the conditions employed. Since release of treated items for unrestricted use is conditional upon full decontamination, the presence of residual explosive contamination on any one item tested will disqualify the entire lot. Subsequent tests with that item and explosive will be performed at more severe treatment conditions. A higher temperature, a longer retention time, or both may be adopted for the next similar test. New test conditions will be established by consultation among TVA, USAEC, USADCS, HWAAP and DZB.

An apparent failure to decontaminate will also be studied for other contributing factors. The most likely ones known at this time are variations in the actual temperature at and near the item(s) and any impediment to the circulation of hot gas over the item. Such factors would be abated if possible. The temperature

control point for the chamber might be relocated to the area most difficult to heat, if it were different from the location established before testing with explosives. A location with insufficient circulation of hot gas to reach treatment temperature or to carry away volatilized explosives could most simply be corrected by changing placement of contaminated items to avoid utilization of the ineffective area.

A finding of redeposition of explosives upon facility surfaces would likely be addressed by instituting hot gas decontamination of the chamber itself. Between tests with contaminated items, the chamber would be heated to the maximum attainable temperature and held for a long enough time to heat the affected surfaces to effective treatment temperature. This measure is scheduled for precautionary application after each ten test runs in order to minimize the chance of deposition of explosive on chamber insulation.

It should be noted that the only corrective measure for inadequate decontamination that is authorized for implementation by test staff is an incremental increase in treatment conditions of time and/or temperature. Any of the efforts toward a procedural correction of failure to decontaminate described above; a change in temperature control point, rearrangement of munitions items, a frequent chamber decontamination cycle, or any other measure of similar scale would require the consultation and concurrence of all participating agencies and organizations. Engineering changes to the facility itself are beyond the scope of this test program. If required they would require design and implementation by appropriate parties. The test program would be considered for resumption from the start of facility testing with the HGD system in a new configuration.

#### **3.4.7 Defining Effective Decontamination**

In advance of conducting the test program, consideration must be given as to how the test results may be later viewed and interpreted. In formulating the test plan, it was determined that it would be essential for every spiked projectile or surface area to be tracked for temperature by its own thermocouple. One reason is that if a particular test produced some samples with measurable explosive, and others without, a knowledge of the temperature profile for each test point would be vital in determining and understanding the effects of the process.

Additionally, and equally important, it is considered much more valid from a scientific and engineering perspective to define "effective decontamination" in terms of the conditions an individual item must undergo, as opposed to using the

overall chamber conditions (i.e., a thermocouple reading that tracks the ambient conditions within the chamber). Under a given set of chamber conditions, there would be no guarantee that a sufficient temperature and time would be applied to every item in the chamber, for all types of items or arrangements of items. Furthermore, any deviation in the mechanical operation of the chamber from the original test, such as an increase of air leakage into the chamber, or the construction of a new chamber of different design, could be expected to produce different conditions within the items themselves, even though the same chamber conditions were used.

Instead, by defining effective decontamination in terms of the necessary conditions each individual item must undergo, a proper basis for chamber design and chamber operation is formed. This basis then becomes universally applicable. For the chamber operator, this basis becomes a goal to be achieved throughout the chamber for all items (which could be verified by a network of thermocouples, for instance). With experience, this would also aid the operator in learning what arrangements of items are best to achieve the complete decontamination of all items. For the chamber designer, this basis can be used to produce the design of an efficient, uniformly heating chamber in Phase II of this program.

### **3.5**

#### **Other Activities**

#### **3.5.1**

##### **Activated Carbon**

At the request of DZB's environmental officer, a sample of activated carbon from the HWAAP water treatment plant was delivered to TVA. It contains explosive compounds adsorbed from plant aqueous effluent. The explosive content is estimated at 8 percent, which is below the level of 10 percent that is considered to be the minimum capable of presenting a fire or explosion risk. However, it is considered a hazardous waste and must be disposed of as such at considerable cost. DZB requests that the carbon be treated under HGD conditions to study the possibility of regenerating it for reuse on site or at least decontaminating it for disposal as solid waste. If practicable, this approach would provide cost savings of up to \$200,000 per year at HWAAP.

Gram quantities of carbon will be treated under typical HGD conditions in a laboratory furnace, extracted, and analyzed for explosive residue. If successful, the method will be considered for future larger scale testing.

### 3.5.2

#### Other Items and Explosives

No other explosive compounds or munition items have as yet been identified as candidates for HGD treatment.

The possibility of decontaminating explosive or munitions plant process equipment for safe and economical maintenance has been discussed. At present, plant equipment must be manually dismantled and cleaned of explosive residue before repairs can be undertaken. Some equipment is discarded because it would cost more to decontaminate and repair than to replace. If the HGD process is shown to be satisfactory for munitions decontamination, tests will be proposed for study of the application of HGD to plant equipment. The effect of the temperatures required on working parts, the ability of hot gas to remove explosive residues from the typically more complex shapes of equipment, the degree of preliminary dismantlement required, and the best means of handling nonmetallic components should all be investigated.

This test program is limited to the items and explosives described in the scope of work and test plan. Trials of materials from other installations which are potential users of HGD would be considered for future work. Suitability and availability of the HWAAP/WADF facility and concurrence of involved agencies would be required.

Production Mode Operations. As mentioned in 3.1 above, prior tests of the HGD process involved treatment of one or a few items per lot. This test program expands the quantity of items to be tested to the number that can conveniently be accommodated on the chamber's narrow-gauge railcars, placed in racks that allow free circulation of hot gas for heating and vapor dispersal. Only for a few items, e.g., 175mm projectiles, does the test lading approach the car's weight capacity. More elaborate racks and longer loading and unloading time would be required for most other items to attain either the maximum weight for the car or the maximum height and width that would enter the chamber on the car, while maintaining separation of items for hot gas circulation. Even that measure has been predicted to limit HGD chamber capacity to less than the upstream stages of the demilitarization process. In the interest of increasing chamber throughput and avoiding a plant bottleneck, HWAAP/DZB propose to adopt a bulk lading of the chamber. Items such as projectiles and segments of other munitions might be handled in box, basket, or cage type containers. Larger items might be palletized or simply stacked up. The railcars would not be used, items would be moved in and out of the chamber by forklift.



The application of test data obtained from specific arrangement of items in the chamber to operations with such bulk material handling may be questionable. Stacking or random piling of items would be expected to impede gas circulation. This, in combination of the greater thermal mass of the bulk lading would increase heatup and cooldown times. Items on the inside of a container or stack might not reach effective treatment temperature in any reasonable time. Blockage of gas flow over individual items could well prevent the dispersal of volatilized explosives to the chamber exhaust. The concrete floor of the chamber can serve as a heat sink which would cause items stacked directly on the floor to remain at a lower temperature than desired.

The magnitude of these effects cannot be estimated on the basis of present information, nor on the results of tests with items specifically arranged as shown in this plan. Therefore, at the conclusion of these tests with racked munitions, a supplementary test (Test #31) will be made with the chamber filled to capacity with an item and arrangement as selected by HWAAP/DZB. The temperature of as many items as possible will be monitored, and process samples will be taken from the maximum number of items as in any prior test.

Periodic gas samples will track the volatilization of explosives. Initial tests will have one gas sample taken starting one hour after achieving nominal chamber operating temperature and one taken late in the test, e.g., the fifth hour of a six-hour test. The gas sampling schedule may be revised based on experience by the time of Test #31 in a high-capacity lading simulating production mode.

The nominal treatment temperature and time for high-capacity lading will be the values established for that item and explosive during tests with items in fixtures. Attainment of the desired temperature will have to be more closely monitored and evaluated. It is expected that temperature throughout the chamber is likely to vary more widely and take longer to reach a steady state than when fixtures are used.

It is further recommended that any reserve capability of time, funds, supplies, and other resources be applied to additional tests to simulate the proposed operational mode. The need for extended testing in production mode would be considered after those trials. Such tests would constitute either a revision and extension of the scope of this test program or a complete new test of the HGD facility in production mode.

## 4.0 TEST RESULTS AND REPORTS

### 4.1 Coverage

At the conclusion of the test program described in this test plan, TVA will prepare a technical report and submit it to USAEC. The report will cover all test activities under the current plan plus any other test activities that may arise from mid-course plan changes. If, at the completion of the test plan, additional test work is found to be needed and is requested by USAEC, an interim report covering all work to date can be prepared and issued if so desired by USAEC.

The purpose of the technical report will be to communicate: (1) all test, sampling, and analytical procedures in sufficient detail such that the USAEC or any other technically skilled organization could repeat the procedures; (2) a full and detailed accounting of all test results; (3) all conclusions that can be derived from the results; and (4) recommendations for further test work.

Specific points to be investigated in the test work and answered in the report include:

- The criteria by which the treatment method is evaluated--generally, all criteria are considered unacceptable that attempt to use statistical methods to correlate test results and thereby "predict" a set of test conditions (time, temperature, etc.) under which the quantity of residual explosive will become zero. Criteria must be developed that, to the extent possible, offer direct evidence that the test chamber conditions are sufficient to completely remove all explosive from all the test items.
- Application to production mode--determine the general applicability of the method to production conditions, i.e., the method's applicability to "random" or "piled" arrangements of items in the test chamber, unlike the ordered item arrangements required by the formal test plan.

The report will include, but is not limited to, the following topics and features:

- Methods development--will cover all specialized laboratory or test methods that were developed to facilitate the test plan. A description of the manner in which each new method was developed and the rationale for its efficacy will be included.

- Test procedures used in the field.
- Spiking, sampling, and analytical procedures.
- QA/QC methods used in the field and in the analytical laboratory.
- QA/QC results from field sampling and laboratory analysis.
- Test results--data evaluation, graphical and tabular presentation of data.
- Photographs--included as necessary to enhance understanding of the equipment arrangement, the test and sampling procedures, the arrangement of test items, and where possible, the visual evidence of the test results (such as an explosive color indicator).
- Conclusions
- Recommendations.
- Raw data--in appendix or under separate cover.

## 4.2

### Format

The report will be presented in a standard technical format, with USAEC's official front cover and "Report Documentation Page" for identification. The report sections and subsections will be designated numerically (as is done in this test plan) in order to facilitate referencing of the report in oral and written communications.

Tentatively, the report sections to be presented are:

INTRODUCTION

SUMMARY

METHODS DEVELOPMENT

TEST PROCEDURES

ANALYTICAL PROCEDURES

RESULTS

CONCLUSIONS AND RECOMMENDATIONS

## **APPENDIX A**

### **LABORATORY PROTOCOL**

## LABORATORY PROTOCOL

1.0	Introduction . . . . .	A-1
2.0	General Information . . . . .	A-1
3.0	Analytical Procedures and Calibration . . . . .	A-2
4.0	Data Reduction, Validation, and Reporting . . . . .	A-4
5.0	Internal Quality Control Checks . . . . .	A-5
6.0	Performance and System Audits . . . . .	A-8
7.0	Calculation of Data Quality Indicators . . . . .	A-9
8.0	Corrective Action . . . . .	A-10
9.0	Quality Control Reports to Management . . . . .	A-10

## LABORATORY PROTOCOL

### 1.0 Introduction

The Analytical Laboratory of Support Services (ALSS), Tennessee Valley Authority, Resource Group located in Muscle Shoals, Alabama will provide analytical chemistry support for a U. S. Army Environmental Center (USUSAEC) project concerning hot gas decontamination of explosives-contaminated munitions. The ALSS will provide laboratory support in two major areas of the project:

1. Methods Development -- development of special sampling and analytical procedures that are required to fulfill the objectives of the project. (Section 2.3 in the body of the main report describes the sub-projects to be undertaken during the methods development work.)
2. Sample Analysis -- analysis of routine samples generated during the HGD chamber tests at HWAAP.

This appendix provides a compilation of work activities, formal procedures, and other details of the work the ALSS will provide in support of the HGD project.

### 2.0 General Information

#### 2.1 Sample Turnaround Time

For the routine samples from the HGD chamber tests, ALSS will be able to provide 48-hour turnaround time for analytical data. Complete data packages should be available one week after receipt of the samples. For samples that require dilution or cleanup, ALSS should be able to provide seven-day turnaround time for analytical data after receipt of samples. Data packages will then be available fourteen days after receipt of samples.

#### 2.2 Project Organization and Responsibilities

The laboratory manager provides project oversight and is responsible for final data integrity. The laboratory manager is responsible for providing monthly ALSS project reports to USUSAEC, through TVA's project management staff.

The Quality Assurance Officer of ALSS reports directly to the laboratory manager and has no direct responsibilities in testing or analysis of the samples. The QA Officer is responsible for auditing actions and documentation to ensure adherence to this laboratory protocol. The QA Officer is responsible for providing quarterly quality control data reports to the laboratory manager.

Research Chemists are responsible for planning, designing, testing, and documenting the various sub-projects assigned to them. They are responsible for producing periodic progress reports to the laboratory manager. They are responsible for review of data falling under their areas of responsibility.

Chemical Laboratory Analysts are responsible for following procedures and instructions to provide analytical measurements required in the course of the project. They are responsible for review of the data they produce, documentation of analytical runs, and equipment maintenance.

## **2.3**

### **Research Records**

Data logging - Suitable records will be maintained as bound research logbooks, instrument logs, worksheets, machine printouts, chromatograms, plots, and case narratives to be able to completely reconstruct all assessments, decisions, quantitative data measurements, preparation of standard solutions, use of standard solutions, and preparation of quality control samples. Components of each analytical run should be preserved including calibration printouts and printouts of rejected samples not included in final data packages.

Written procedures produced as a result of this project will completely describe all actions required to perform the work correctly and to assess the quality of the work.

Nonconformances and problems identified in the course of this project will be documented, corrected, and tracked to conclusion in a rigorous and controlled manner.

## **2.4**

### **Sample Custody**

All test samples from the HGD chamber tests will be handled in accordance with ALSS procedure SP-0001, "Sample Chain of Custody."

### **3.0 Analytical Procedures and Calibration**

#### **3.1 EPA-Approved or Other Validated Standard Methods**

The procedures developed under the methods development phase of the project will be utilized for all sampling and analysis work. Precision and accuracy of the procedures will be demonstrated before they are used for analysis of samples.

#### **3.2 Nonstandard or Modified Methods**

Any modifications to Method 8330 or the Ammonium Picrate Method that are found to be necessary to maintain the efficacy of the methods will be documented. Any modifications found to be necessary after the start of the HGD chamber tests will be reviewed, approved, and promulgated to those performing the work as written procedures in accordance with ALSS Procedure GLP-0001 "Laboratory Procedure Preparation" and GLP-0003, "Procedure Preparation and Distribution."

#### **3.3 Calibration Procedures and Frequency**

The calibration frequencies and tests set forth in Method 8330 will be the guidelines for calibration of the equipment used in the modified methods.

### **4.0 Data Reduction, Validation, and Reporting**

#### **4.1 Data Reduction**

Analytical data will be calculated and reduced on vendor-supplied chromatographic software. If that software is not adequate to perform all calculations, computer spreadsheets or programs will be developed to carry out the computations. These spreadsheets or programs will be documented in accordance with ALSS procedure GLP-0017, "Control of Changes to Software."

Chemical laboratory analysts are responsible for calculation and reduction of data.

#### **4.2 Data Validation**

Group supervisors or team leaders (analytical chemists or research chemists) are responsible for data validation in the methods development phase of the project. Similarly, they are responsible for review and validation of analytical data produced during the analysis of routine test samples.



#### 4.3

##### **Data Reporting**

Analytical data are to be reported in units of micrograms per smear (wipe) or micrograms per liter for liquid samples. The engineers designing field sampling experiments will have the data available on smear areas so that the results may be calculated in micrograms per square centimeter or other appropriate units. (For concentrations greater than 10,000 micrograms, milligrams may be used as the preferred unit.) All areas will be identified in the final report. Instrument detection limits, method detection limits, and sample detection limits will be reported or made available for each run. Surrogate recovery, recovery of matrix spikes, and recovery of quality control samples will be expressed in percent recovered.

All analytical data from a single HGD chamber test will be reported in an analytical data package. Each package will include:

- Sample description or identification information.
- Sample analytical results with surrogate recoveries.
- Quality control sample results with surrogate recoveries and percent recovery of the added compounds.

Sufficient data will be maintained such that every result can be substantiated, if further review is desired. The goal is to maintain records such that every analytical result could be reconstructed and that every decision made during the development of the written procedures could be substantiated.

#### 4.4

##### **Records Retention**

Records of experiments and analyses will be maintained for a period of three years after the end of the project. This will include machine printouts or chromatogram traces, logbooks, notebooks, log-sheets, standard material use logs, raw data calculation sheets and the like. Computer media utilized to store analytical file backups or raw data files will be stored for the lifetime of the project plus one year due to the limited lifetime of computer storage media.

Due to safety considerations, no attempt will be made to store samples or sample extracts beyond that period of time required for initial assessment and review of laboratory data.

Data from failed attempts at analysis will be maintained along with supporting documentation.

#### 4.5

##### Data Qualification Codes

Abbreviation codes which may appear in the analytical data packages include:

SM - Surrogate recovery out of limits. Matrix effect suspected.

SD - Surrogate recovery low due to dilution. (Analyte concentration was so high that the sample had to be diluted to be analyzed.)

NA - Compound Not Analyzed

ND - Compound not detected (analysis value falls below the method detection limit (MDL))

TR or Trace - Compound present at trace level (i.e., there was an indication of the compound, but the concentration was less than the MDL and was not quantifiable.)

MX - Matrix spike or matrix spike duplicate recovery was outside limits due to suspected matrix effects.

#### 5.0

##### Internal Quality Control Checks

Under the requirements of Chapters 1 and 4 of SW-846 and the further specific requirements of Method 8330, a variety of quality control samples will be run.

#### 5.1

##### Setup QC

The following quality control checks are specified in Method 8330 and Method 8000, Chapters 7 and 8, as referenced by Method 8330.

#### 5.1.1

##### General

ALSS will demonstrate that all glassware and reagents are free of interferences by running blank samples. Blanks should include acetonitrile, water, methanol, or any other solvents used in the process.

ALSS will run a QC check sample set of known concentration to ensure method precision and accuracy are defined.

Retention time windows for laboratory analysis equipment will be established.

Each analyst must demonstrate the ability to generate acceptable results with the methods.

#### **5.1.2      Method Detection Limits**

ALSS will determine method detection limits as defined in 40 CFR Part 136, Appendix B, Revision 1.11.

#### **5.1.3      Retention Time Windows**

Three injections are made of each analyte during a 72-hour period, and the retention times are determined for each injection. The means and standard deviations of the retention time data are calculated. Plus or minus three standard deviations from the mean value is to be used as the retention time window for each analyte. (Reference: Section 7.5 of Method 8000A.) When a new column is installed, retention time windows must be redetermined.

#### **5.1.4      Method Accuracy and Precision**

A quality control check sample, which will have been produced independently from the calibration standards and which contains each analyte, will be analyzed four times. The average recovery and the standard deviation will be calculated for each analyte. If correctly analyzed, the analysis values should compare well to those listed in Tables 3, 4, 5, 6, and 7 of Method 8330 for a similar matrix. Any analyte which falls outside limits will be analyzed again in a similar manner after problems are resolved. (Reference: Section 8.6 of Method 8000A.)

### **5.2      Calibration QC**

#### **5.2.1      Method 8000A/8330 Calibration QC**

Reference: Method 8000A Section 7.4 and Method 8330 Section 7.3

Calibration will be performed in triplicate with standards of five concentrations over the range of interest or range of linear response of the device. The lowest concentration should be approximately equal to the method detection limit.

At the beginning of each day, the midpoint calibration standard will be analyzed in triplicate. The response factor for the average of these three points must be within 15 percent of the response factor for the initial calibration. If not, the machine will be recalibrated. Then at least every ten samples and at the end of the run, a single midpoint calibration standard will be run. The response factors for these must be within 15 percent of the mean daily initial response factor. If a midpoint calibration check falls outside the 15 percent limits, all samples since the last valid calibration check will be reanalyzed.

A daily retention time window will be calculated for each analyte using the mean retention time from the initial midpoint calibration standard plus or minus three standard deviations, as determined in the set-up QC section. If the retention time for any analyte from subsequent midpoint calibration standards falls outside the window, those samples analyzed following that midpoint calibration standard must be reanalyzed after the problem is resolved.

#### **5.2.2 Alternate Calibration for Unstable Operating Conditions**

Calibration will be performed with standards of five concentrations at the beginning of each day, run singly. A midpoint calibration standard will be run at least every 10 samples and at the end of the run throughout the day. Any group of ten samples following a midpoint calibration check which falls outside the 15 percent limits will be reanalyzed.

### **5.3 Batch QC**

#### **5.3.1 Definitions**

Batch - A group of no more than 20 samples of the same matrix prepared or extracted at the same time with the same reagents.

Note: If ALSS determines that extracts of smears (wipe samples) are similar in behavior to extract samples, they may be counted as the same matrix for determining batch break points.

Method Blank - A sample of clean reagent carried through preparation and extraction in the same manner as samples.

Surrogates - Chemicals not expected to be present in the samples to be analyzed but with chemical composition and behavior similar to the analytes under consideration. Surrogates are added before preparation and extraction to each test sample and quality control sample in a batch. Surrogate recovery is used to assess matrix effects and to monitor the performance of the extraction and analytical system.

Matrix Spike - An aliquot of a sample spiked with a known concentration of all target analytes. Spike concentration is set to read at five times the method quantitation limit in the sample. One matrix spike is run for each batch. Spiking occurs prior to sample preparation and analysis.

Matrix Spike Duplicate - A second aliquot of the same sample treated as the matrix spike.

Quality Control Check Sample - A sample containing mid-range concentrations of analytes of interest with concentrations known to the analyst. This sample is made from a separate stock of standard material than calibration and spiking solutions when possible.

Duplicate - A second aliquot of a sample.

### **5.3.2 Batch QC Samples**

Surrogates will be added to each sample, blank, and quality control sample before extraction or preparation.

One matrix spike will be run with each batch.

One duplicate or matrix spike duplicate will be run with each batch.

One method blank will be run with each batch.

One quality control check sample will be run with each batch.

Whenever the sampling organization in the field submits additional samples as quality control checks (as is defined in the sampling plan of the test program, contained in the body of this report), ALSS will count these as samples in determining batch size.

## **6.0 Performance and System Audits**

The USAEC, at their option, may include quality control samples as performance audits with any sample set.

QA Audits, site inspections, surveillances, or performance evaluations (crosscheck samples) may be performed by USAEC at any time during the course of the project.

The ALSS Quality Assurance Officer may introduce unknown quality control samples at a suitable frequency, provided reference material is available for constructing the samples.

The ALSS Quality Assurance Officer will periodically inspect logs, records, printouts, results of quality control checks, documentation, case narratives, research notebooks, and other quality related aspects of the project to ensure detailed compliance is in effect. Results of these inspections or internal audits will be reported in writing to the Laboratory Manager. Nonconformances will be documented and tracked in accordance with ALSS Procedure GLP-0005, "Nonconformances and Corrective Actions."

## **7.0      Calculation of Data Quality Indicators**

### **7.1      Common Data Quality Indicators**

Relative percent difference, standard deviation, accuracy, completeness, and other commonly used statistical indicators are to be calculated as defined in Chapter 1 of SW-846, 3rd Edition.

Method Detection Limits will be calculated as defined in 40 CFR 136, Appendix B.

Method Quantitation Limits are defined as five times the Method Detection Limit as in 40 CFR 136, Appendix B.

### **7.2      Project-Specific Indicators**

Mass balance for a series of extractions will be calculated as follows:

Mass Balance = Mass out / Mass in

Where "Mass in" is the mass in milligrams spiked onto a clean surface and "Mass out" is defined as

Mass out =  $C_1 \cdot V_1 + C_2 \cdot V_2 + C_3 \cdot V_3 \dots$

Here  $C_1$  is the concentration from the first extraction in mg/L

$V_1$  is the volume in the first extraction in L

and so forth.

The uncertainty in the mass balance is equal to the square root of the sum of similar terms as in the following equation:

$$T_i = (C_i \cdot V_i)^2 [(\Delta C_i / C_i)^2 + (\Delta V_i / V_i)^2]$$

Where  $\Delta C_i$  is the uncertainty in the concentration  $C_i$  and  $\Delta V_i$  is the uncertainty in the volume  $V_i$ .

So that

$$\text{Uncertainty in Mass Balance} = (T_1 + T_2 + T_3 \dots)^{1/2}$$

## 8.0 Corrective Action

Corrective actions arising from nonconformances determined in the course of audits or analysis of performance evaluation samples will be documented and tracked as described in GLP-0005.

## 9.0 Quality Control Reports to Management

A quarterly quality control data report will be written by the ALSS QA Officer addressing:

- Changes in the laboratory's portion of the QA Project plan.
- Changes in analytical procedures.
- Summary of QC program results, summary of training, summary of accomplishments.
- Results of audits, results of performance sample evaluations, and any significant problems with problem resolutions.
- Data quality assessment in terms of precision, accuracy, completeness, and MDL.
- Discussion of whether QA objectives were met.

## **APPENDIX B**

### **METHODS AND PROCEDURES**



## METHODS AND PROCEDURES

- B1      Quality Assurance
- B2      Organic Analytes
- B3      Gas Chromatography
- B4      Method 8330 Explosives Analysis
- B5      Ammonium Picrate Analysis
- B6      EPA Modified Method 5 Sampling Train
- B7      EPA Wipe Sampling Techniques

**APPENDIX B1**

**QUALITY ASSURANCE**

CHAPTER ONE  
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## CHAPTER ONE QUALITY CONTROL

### 1.0 INTRODUCTION

It is the goal of the U.S. Environmental Protection Agency's (EPA's) quality assurance (QA) program to ensure that all data be scientifically valid, defensible, and of known precision and accuracy. The data should be of sufficient known quality to withstand scientific and legal challenge relative to the use for which the data are obtained. The QA program is management's tool for achieving this goal.

For RCRA analyses, the recommended minimum requirements for a QA program and the associated quality control (QC) procedures are provided in this chapter.

The data acquired from QC procedures are used to estimate the quality of analytical data, to determine the need for corrective action in response to identified deficiencies, and to interpret results after corrective action procedures are implemented. Method-specific QC procedures are incorporated in the individual methods since they are not applied universally.

A total program to generate data of acceptable quality should include both a QA component, which encompasses the management procedures and controls, as well as an operational day-to-day QC component. This chapter defines fundamental elements of such a data collection program. Data collection efforts involve:

1. design of a project plan to achieve the data quality objectives (DQOs);
2. implementation of the project plan; and
3. assessment of the data to determine if the DQOs are met.

The project plan may be a sampling and analysis plan or a waste analysis plan if it covers the QA/QC goals of the Chapter, or it may be a Quality Assurance Project Plan as described later in this chapter.

This chapter identifies the minimal QC components that should be used in the performance of sampling and analyses, including the QC information which should be documented. Guidance is provided to construct QA programs for field and laboratory work conducted in support of the RCRA program.

### 2.0 QA PROJECT PLAN

It is recommended that all projects which generate environment-related data in support of RCRA have a QA Project Plan (QAPjP) or equivalent. In some instances, a sampling and analysis plan or a waste analysis plan may be equivalent if it covers all of the QA/QC goals outlined in this chapter. In addition, a separate QAPjP need not be prepared for routine analyses or activities where the procedures to be followed are described in a Standard

Operating Procedures manual or similar document and include the elements of a QAPjP. These documents should be available and referenced in the documentation and/or records for the analysis activities. The term "QAPjP" in this chapter refers to any of these QA/QC documents.

The QAPjP should detail the QA/QC goals and protocols for a specific data collection activity. The QAPjP sets forth a plan for sampling and analysis activities that will generate data of a quality commensurate with their intended use. QAPjP elements should include a description of the project and its objectives; a statement of the DQOs of the project; identification of those involved in the data collection and their responsibilities and authorities; reference to (or inclusion of) the specific sample collection and analysis procedures that will be followed for all aspects of the project; enumeration of QC procedures to be followed; and descriptions of all project documentation. Additional elements should be included in the QAPjP if needed to address all quality related aspects of the data collection project. Elements should be omitted only when they are inappropriate for the project or when absence of those elements will not affect the quality of data obtained for the project (see reference 1).

The role and importance of DQOs and project documentation are discussed below in Sections 2.1 through 2.6. Management and organization play a critical role in determining the effectiveness of a QA/QC program and ensuring that all required procedures are followed. Section 2.7 discusses the elements of an organization's QA program that have been found to ensure an effective program. Field operations and laboratory operations (along with applicable QC procedures) are discussed in Sections 3 and 4, respectively.

## 2.1 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) for the data collection activity describe the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data. This uncertainty is used to specify the quality of the measurement data required, usually in terms of objectives for precision, bias, representativeness, comparability and completeness. The DQOs should be defined prior to the initiation of the field and laboratory work. The field and laboratory organizations performing the work should be aware of the DQOs so that their personnel may make informed decisions during the course of the project to attain those DQOs. More detailed information on DQOs is available from the U.S. EPA Quality Assurance Management Staff (QAMS) (see references 2 and 4).

## 2.2 PROJECT OBJECTIVES

A statement of the project objectives and how the objectives are to be attained should be concisely stated and sufficiently detailed to permit clear understanding by all parties involved in the data collection effort. This includes a statement of what problem is to be solved and the information required

in the process. It also includes appropriate statements of the DQOs (i.e., the acceptable level of uncertainty in the information).

## 2.3 SAMPLE COLLECTION

Sampling procedures, locations, equipment, and sample preservation and handling requirements should be specified in the QAPjP. Further details on quality assurance procedures for field operations are described in Section 3 of this chapter. The OSW is developing policies and procedures for sampling in a planned revision of Chapter Nine of this manual. Specific procedures for groundwater sampling are provided in Chapter Eleven of this manual.

## 2.4 ANALYSIS AND TESTING

Analytes and properties of concern, analytical and testing procedures to be employed, required detection limits, and requirements for precision and bias should be specified. All applicable regulatory requirements and the project DQOs should be considered when developing the specifications. Further details on the procedures for analytical operations are described in Section 4 of this chapter.

## 2.5 QUALITY CONTROL

The quality assurance program should address both field and laboratory activities. Quality control procedures should be specified for estimating the precision and bias of the data. Recommended minimum requirements for QC samples have been established by EPA and should be met in order to satisfy recommended minimum criteria for acceptable data quality. Further details on procedures for field and laboratory operations are described in Sections 3 and 4, respectively, of this chapter.

## 2.6 PROJECT DOCUMENTATION

Documents should be prepared and maintained in conjunction with the data collection effort. Project documentation should be sufficient to allow review of all aspects of the work being performed. The QAPjP discussed in Sections 3 and 4 is one important document that should be maintained.

The length of storage time for project records should comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent. It is recommended that documentation be stored for three years from submission of the project final report.

Documentation should be secured in a facility that adequately addresses/minimizes its deterioration for the length of time that it is to be retained. A system allowing for the expedient retrieval of information should exist.

Access to archived information should be controlled to maintain the integrity of the data. Procedures should be developed to identify those individuals with access to the data.

## 2.7 ORGANIZATION PERFORMING FIELD OR LABORATORY OPERATIONS

Proper design and structure of the organization facilitates effective and efficient transfer of information and helps to prevent important procedures from being overlooked.

The organizational structure, functional responsibilities, levels of authority, job descriptions, and lines of communication for all project activities should be established and documented. One person may cover more than one organizational function. Each project participant should have a clear understanding of his or her duties and responsibilities and the relationship of those responsibilities to the overall data collection effort.

The management of each organization participating in a project involving data collection activities should establish that organization's operational and QA policies. This information should be documented in the QAPjP. The management should ensure that (1) the appropriate methodologies are followed as documented in the QAPjPs; (2) personnel clearly understand their duties and responsibilities; (3) each staff member has access to appropriate project documents; (4) any deviations from the QAPjP are communicated to the project management and documented; and (5) communication occurs between the field, laboratory, and project management, as specified in the QAPjP. In addition, each organization should ensure that their activities do not increase the risk to humans or the environment at or about the project location. Certain projects may require specific policies or a Health and Safety Plan to provide this assurance.

The management of the participating field or laboratory organization should establish personnel qualifications and training requirements for the project. Each person participating in the project should have the education, training, technical knowledge, and experience, or a combination thereof, to enable that individual to perform assigned functions. Training should be provided for each staff member as necessary to perform their functions properly. Personnel qualifications should be documented in terms of education, experience, and training, and periodically reviewed to ensure adequacy to current responsibilities.

Each participating field organization or laboratory organization should have a designated QA function (i.e., a team or individual trained in QA) to monitor operations to ensure that the equipment, personnel, activities, procedures, and documentation conform with the QAPjP. To the extent possible, the QA monitoring function should be entirely separate from, and independent of, personnel engaged in the work being monitored. The QA function should be responsible for the QA review.



### 2.7.1 Performance Evaluation

Performance evaluation studies are used to measure the performance of the laboratory on unknown samples. Performance evaluation samples are typically submitted to the laboratory as blind samples by an independent outside source. The results are compared to predetermined acceptance limits. Performance evaluation samples can also be submitted to the laboratory as part of the QA function during internal assessment of laboratory performance. Records of all performance evaluation studies should be maintained by the laboratory. Problems identified through participation in performance evaluation studies should be immediately investigated and corrected.

### 2.7.2 Internal Assessment by QA Function

Personnel performing field and laboratory activities are responsible for continually monitoring individual compliance with the QAPjP. The QA function should review procedures, results and calculations to determine compliance with the QAPjP. The results of this internal assessment should be reported to management with requirements for a plan to correct observed deficiencies.

### 2.7.3 External Assessment

The field and laboratory activities may be reviewed by personnel external to the organization. Such an assessment is an extremely valuable method for identifying overlooked problems. The results of the external assessment should be submitted to management with requirements for a plan to correct observed deficiencies.

### 2.7.4 On-Site Evaluation

On-site evaluations may be conducted as part of both internal and external assessments. The focus of an on-site evaluation is to evaluate the degree of conformance of project activities with the applicable QAPjP. On-site evaluations may include, but are not limited to, a complete review of facilities, staff, training, instrumentation, procedures, methods, sample collection, analyses, QA policies and procedures related to the generation of environmental data. Records of each evaluation should include the date of the evaluation, location, the areas reviewed, the person performing the evaluation, findings and problems, and actions recommended and taken to resolve problems. Any problems identified that are likely to affect data integrity should be brought immediately to the attention of management.

#### 2.7.4.1 Field Activities

The review of field activities should be conducted by one or more persons knowledgeable in the activities being reviewed and include evaluating, at a minimum, the following subjects:

Completeness of Field Reports -- This review determines whether all requirements for field activities in the QAPjP have been fulfilled, that complete records exist for each field activity, and that the procedures

specified in the QAPjP have been implemented. Emphasis on field documentation will help assure sample integrity and sufficient technical information to recreate each field event. The results of this completeness check should be documented, and environmental data affected by incomplete records should be identified.

Identification of Valid Samples -- This review involves interpretation and evaluation of the field records to detect problems affecting the representativeness of environmental samples. Examples of items that might indicate potentially invalid samples include improper well development, improperly screened wells, instability of pH or conductivity, and collection of volatiles near internal combustion engines. The field records should be evaluated against the QAPjP and SOPs. The reviewer should document the sample validity and identify the environmental data associated with any poor or incorrect field work.

Correlation of Field Test Data -- This review involves comparing any available results of field measurements obtained by more than one method. For example, surface geophysical methods should correlate with direct methods of site geologic characterization such as lithologic logs constructed during drilling operations.

Identification of Anomalous Field Test Data -- This review identifies any anomalous field test data. For example, a water temperature for one well that is 5 degrees higher than any other well temperature in the same aquifer should be noted. The reviewer should evaluate the impact of anomalous field measurement results on the associated environmental data.

Validation of Field Analyses -- This review validates and documents all data from field analysis that are generated in situ or from a mobile laboratory as specified in Section 2.7.4.2. The reviewer should document whether the QC checks meet the acceptance criteria, and whether corrective actions were taken for any analysis performed when acceptance criteria were exceeded.

#### 2.7.4.2 Laboratory Activities

The review of laboratory data should be conducted by one or more persons knowledgeable in laboratory activities and include evaluating, at a minimum, the following subjects:

Completeness of Laboratory Records -- This review determines whether: (1) all samples and analyses required by the QAPjP have been processed, (2) complete records exist for each analysis and the associated QC samples, and that (3) the procedures specified in the QAPjP have been implemented. The results of the completeness check should be documented, and environmental data affected by incomplete records should be identified.

Evaluation of Data with Respect to Detection and Quantitation Limits -- This review compares analytical results to required quantitation limits. Reviewers should document instances where detection or quantitation limits

exceed regulatory limits, action levels, or target concentrations specified in the QAPjP.

Evaluation of Data with Respect to Control Limits -- This review compares the results of QC and calibration check samples to control criteria. Corrective action should be implemented for data not within control limits. The reviewer should check that corrective action reports, and the results of reanalysis, are available. The review should determine whether samples associated with out-of-control QC data are identified in a written record of the data review, and whether an assessment of the utility of such analytical results is recorded.

Review of Holding Time Data -- This review compares sample holding times to those required by the QAPjP, and notes all deviations.

Review of Performance Evaluation (PE) Results -- PE study results can be helpful in evaluating the impact of out-of-control conditions. This review documents any recurring trends or problems evident in PE studies and evaluates their effect on environmental data.

Correlation of Laboratory Data -- This review determines whether the results of data obtained from related laboratory tests, e.g., Purgeable Organic Halides (POX) and Volatile Organics, are documented, and whether the significance of any differences is discussed in the reports.

#### 2.7.5 QA Reports

There should be periodic reporting of pertinent QA/QC information to the project management to allow assessment of the overall effectiveness of the QA program. There are three major types of QA reports to project management:

Periodic Report on Key QA Activities -- Provides summary of key QA activities during the period, stressing measures that are being taken to improve data quality; describes significant quality problems observed and corrective actions taken; reports information regarding any changes in certification/accreditation status; describes involvement in resolution of quality issues with clients or agencies; reports any QA organizational changes; and provides notice of the distribution of revised documents controlled by the QA organization (i.e., procedures).

Report on Measurement Quality Indicators -- Includes the assessment of QC data gathered over the period, the frequency of analyses repeated due to unacceptable QC performance, and, if possible, the reason for the unacceptable performance and corrective action taken.

Reports on QA Assessments -- Includes the results of the assessments and the plan for correcting identified deficiencies; submitted immediately following any internal or external on-site evaluation or upon receipt of the results of any performance evaluation studies.

### 3.0 FIELD OPERATIONS

The field operations should be conducted in such a way as to provide reliable information that meets the DQOs. To achieve this, certain minimal policies and procedures should be implemented. The OSW is considering revisions of Chapter Nine and Eleven of this manual. Supplemental information and guidance is available in the RCRA Ground-Water Monitoring Technical Enforcement Guidance Document (TEGD) (Reference 3). The project documentation should contain the information specified below.

#### 3.1 FIELD LOGISTICS

The QAPjP should describe the type(s) of field operations to be performed and the appropriate area(s) in which to perform the work. The QAPjP should address ventilation, protection from extreme weather and temperatures, access to stable power, and provision for water and gases of required purity.

Whenever practical, the sampling site facilities should be examined prior to the start of work to ensure that all required items are available. The actual area of sampling should be examined to ensure that trucks, drilling equipment, and personnel have adequate access to the site.

The determination as to whether sample shipping is necessary should be made during planning for the project. This need is established by evaluating the analyses to be performed, sample holding times, and location of the site and the laboratory. Shipping or transporting of samples to a laboratory should be done within a timeframe such that recommended holding times are met.

Samples should be packaged, labelled, preserved (e.g., preservative added, iced, etc.), and documented in an area which is free of contamination and provides for secure storage. The level of custody and whether sample storage is needed should be addressed in the QAPjP.

Storage areas for solvents, reagents, standards, and reference materials should be adequate to preserve their identity, concentration, purity, and stability prior to use.

Decontamination of sampling equipment may be performed at the location where sampling occurs, prior to going to the sampling site, or in designated areas near the sampling site. Project documentation should specify where and how this work is accomplished. If decontamination is to be done at the site, water and solvents of appropriate purity should be available. The method of accomplishing decontamination, including the required materials, solvents, and water purity should be specified.

During the sampling process and during on-site or in situ analyses, waste materials are sometimes generated. The method for storage and disposal of these waste materials that complies with applicable local, state and Federal regulations should be specified. Adequate facilities should be provided for the collection and storage of all wastes, and these facilities should be operated so

as to minimize environmental contamination. Waste storage and disposal facilities should comply with applicable federal, state, and local regulations.

The location of long-term and short-term storage for field records, and the measures to ensure the integrity of the data should be specified.

### 3.2 EQUIPMENT/INSTRUMENTATION

The equipment, instrumentation, and supplies at the sampling site should be specified and should be appropriate to accomplish the activities planned. The equipment and instrumentation should meet the requirements of specifications, methods, and procedures as specified in the QAPjP.

### 3.3 OPERATING PROCEDURES

The QAPjP should describe or make reference to all field activities that may affect data quality. For routinely performed activities, standard operating procedures (SOPs) are often prepared to ensure consistency and to save time and effort in preparing QAPjPs. Any deviation from an established procedure during a data collection activity should be documented. The procedures should be available for the indicated activities, and should include, at a minimum, the information described below.

#### 3.3.1 Sample Management

The numbering and labeling system, chain-of-custody procedures, and how the samples are to be tracked from collection to shipment or receipt by the laboratory should be specified. Sample management procedures should also specify the holding times, volumes of sample required by the laboratory, required preservatives, and shipping requirements.

#### 3.3.2 Reagent/Standard Preparation

The procedures describing how to prepare standards and reagents should be specified. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and record keeping for stocks and dilutions should be included.

#### 3.3.3 Decontamination

The procedures describing decontamination of field equipment before and during the sample collection process should be specified. These procedures should include cleaning materials used, the order of washing and rinsing with the cleaning materials, requirements for protecting or covering cleaned equipment, and procedures for disposing of cleaning materials.

#### 3.3.4 Sample Collection

The procedures describing how the sampling operations are actually performed in the field should be specified. A simple reference to standard methods is not sufficient, unless a procedure is performed exactly as described in the published method. Methods from source documents published by the EPA, American Society for Testing and Materials, U.S. Department of the Interior, National Water Well Association, American Petroleum Institute, or other recognized organizations with appropriate expertise should be used, if possible. The procedures for sample collection should include at least the following:

- Applicability of the procedure,
- Equipment required,
- Detailed description of procedures to be followed in collecting the samples,
- Common problems encountered and corrective actions to be followed, and
- Precautions to be taken.

#### 3.3.5 Field Measurements

The procedures describing all methods used in the field to determine a chemical or physical parameter should be described in detail. The procedures should address criteria from Section 4, as appropriate.

#### 3.3.6 Equipment Calibration And Maintenance

The procedures describing how to ensure that field equipment and instrumentation are in working order should be specified. These describe calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, and service arrangements for equipment. Calibration and maintenance of field equipment and instrumentation should be in accordance with manufacturers' specifications or applicable test specifications and should be documented.

#### 3.3.7 Corrective Action

The procedures describing how to identify and correct deficiencies in the sample collection process should be specified. These should include specific steps to take in correcting deficiencies such as performing additional decontamination of equipment, resampling, or additional training of field personnel. The procedures should specify that each corrective action should be documented with a description of the deficiency and the corrective action taken, and should include the person(s) responsible for implementing the corrective action.

### 3.3.8 Data Reduction and Validation

The procedures describing how to compute results from field measurements and to review and validate these data should be specified. They should include all formulas used to calculate results and procedures used to independently verify that field measurement results are correct.

### 3.3.9 Reporting

The procedures describing the process for reporting the results of field activities should be specified.

### 3.3.10 Records Management

The procedures describing the means for generating, controlling, and archiving project-specific records and field operations records should be specified. These procedures should detail record generation and control and the requirements for record retention, including type, time, security, and retrieval and disposal authorities.

Project-specific records relate to field work performed for a project. These records may include correspondence, chain-of-custody records, field notes, all reports issued as a result of the work, and procedures used.

Field operations records document overall field operations and may include equipment performance and maintenance logs, personnel files, general field procedures, and corrective action reports.

### 3.3.11 Waste Disposal

The procedures describing the methods for disposal of waste materials resulting from field operations should be specified.

## 3.4 FIELD QA AND QC REQUIREMENTS

The QAPjP should describe how the following elements of the field QC program will be implemented.

### 3.4.1 Control Samples

Control samples are QC samples that are introduced into a process to monitor the performance of the system. Control samples, which may include blanks (e.g., trip, equipment, and laboratory), duplicates, spikes, analytical standards, and reference materials, can be used in different phases of the data collection process beginning with sampling and continuing through transportation, storage, and analysis.

Each day of sampling, at least one field duplicate and one equipment rinsate should be collected for each matrix sampled. If this frequency is not appropriate for the sampling equipment and method, then the appropriate changes

should be clearly identified in the QAPjP. When samples are collected for volatile organic analysis, a trip blank is also recommended for each day that samples are collected. In addition, for each sampling batch (20 samples of one matrix type), enough volume should be collected for at least one sample so as to allow the laboratory to prepare one matrix spike and either one matrix duplicate or one matrix spike duplicate for each analytical method employed. This means that the following control samples are recommended:

- Field duplicate (one per day per matrix type)
- Equipment rinsate (one per day per matrix type)
- Trip blank (one per day, volatile organics only)
- Matrix spike (one per batch [20 samples of each matrix type])
- Matrix duplicate or matrix spike duplicate (one per batch)

Additional control samples may be necessary in order to assure data quality to meet the project-specific DQOs.

#### 3.4.2 Acceptance Criteria

Procedures should be in place for establishing acceptance criteria for field activities described in the QAPjP. Acceptance criteria may be qualitative or quantitative. Field events or data that fall outside of established acceptance criteria may indicate a problem with the sampling process that should be investigated.

#### 3.4.3 Deviations

All deviations from plan should be documented as to the extent of, and reason for, the deviation. Any activity not performed in accordance with procedures or QAPjPs is considered a deviation from plan. Deviations from plan may or may not affect data quality.

#### 3.4.4 Corrective Action

Errors, deficiencies, deviations, certain field events, or data that fall outside established acceptance criteria should be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the system. The investigation of the problem and any subsequent corrective action taken should be documented.

#### 3.4.5 Data Handling

All field measurement data should be reduced according to protocols described or referenced in the QAPjP. Computer programs used for data reduction should be validated before use and verified on a regular basis. All information used in the calculations should be recorded to enable reconstruction of the final result at a later date.

Data should be reported in accordance with the requirements of the end-user as described in the QAPjP.



### 3.5 QUALITY ASSURANCE REVIEW

The QA Review consists of internal and external assessments to ensure that QA/QC procedures are in use and to ensure that field staff conform to these procedures. QA review should be conducted as deemed appropriate and necessary.

### 3.6 FIELD RECORDS

Records provide the direct evidence and support for the necessary technical interpretations, judgments, and discussions concerning project activities. These records, particularly those that are anticipated to be used as evidentiary data, should directly support current or ongoing technical studies and activities and should provide the historical evidence needed for later reviews and analyses. Records should be legible, identifiable, and retrievable and protected against damage, deterioration, or loss. The discussion in this section (3.6) outlines recommended procedures for record keeping. Organizations which conduct field sampling should develop appropriate record keeping procedures which satisfy relevant technical and legal requirements.

Field records generally consist of bound field notebooks with prenumbered pages, sample collection forms, personnel qualification and training forms, sample location maps, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and field change request forms. All records should be written in indelible ink.

Procedures for reviewing, approving, and revising field records should be clearly defined, with the lines of authority included. It is recommended that all documentation errors should be corrected by drawing a single line through the error so it remains legible and should be initialed by the responsible individual, along with the date of change. The correction should be written adjacent to the error.

Records should include (but are not limited to) the following:

Calibration Records & Traceability of Standards/Reagents -- Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program should include provisions for documentation of frequency, conditions, standards, and records reflecting the calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used. A program for verifying and documenting the accuracy of all working standards against primary grade standards should be routinely followed.

Sample Collection -- To ensure maximum utility of the sampling effort and resulting data, documentation of the sampling protocol, as performed in the field, is essential. It is recommended that sample collection records contain, at a minimum, the names of persons conducting the activity, sample number, sample location, equipment used, climatic conditions, documentation of adherence to protocol, and unusual observations. The

actual sample collection record is usually one of the following: a bound field notebook with prenumbered pages, a pre-printed form, or digitized information on a computer tape or disc.

Chain-of-Custody Records -- The chain-of-custody involving the possession of samples from the time they are obtained until they are disposed or shipped off-site should be documented as specified in the QAPjP and should include the following information: (1) the project name; (2) signatures of samplers; (3) the sample number, date and time of collection, and grab or composite sample designation; (4) signatures of individuals involved in sample transfer; and (5) if applicable, the air bill or other shipping number.

Maps and Drawings -- Project planning documents and reports often contain maps. The maps are used to document the location of sample collection points and monitoring wells and as a means of presenting environmental data. Information used to prepare maps and drawings is normally obtained through field surveys, property surveys, surveys of monitoring wells, aerial photography or photogrammetric mapping. The final, approved maps and/or drawings should have a revision number and date and should be subject to the same controls as other project records.

QC Samples -- Documentation for generation of QC samples, such as trip and equipment rinsate blanks, duplicate samples, and any field spikes should be maintained.

Deviations -- All deviations from procedural documents and the QAPjP should be recorded in the site logbook.

Reports -- A copy of any report issued and any supporting documentation should be retained.

#### 4.0 LABORATORY OPERATIONS

The laboratory should conduct its operations in such a way as to provide reliable information. To achieve this, certain minimal policies and procedures should be implemented.

##### 4.1 FACILITIES

The QAPjP should address all facility-related issues that may impact project data quality. Each laboratory should be of suitable size and construction to facilitate the proper conduct of the analyses. Adequate bench space or working area per analyst should be provided. The space requirement per analyst depends on the equipment or apparatus that is being utilized, the number of samples that the analyst is expected to handle at any one time, and the number of operations that are to be performed concurrently by a single analyst. Other issues to be considered include, but are not limited to, ventilation, lighting,

control of dust and drafts, protection from extreme temperatures, and access to a source of stable power.

Laboratories should be designed so that there is adequate separation of functions to ensure that no laboratory activity has an adverse effect on the analyses. The laboratory may require specialized facilities such as a perchloric acid hood or glovebox.

Separate space for laboratory operations and appropriate ancillary support should be provided, as needed, for the performance of routine and specialized procedures.

As necessary to ensure secure storage and prevent contamination or misidentification, there should be adequate facilities for receipt and storage of samples. The level of custody required and any special requirements for storage such as refrigeration should be described in planning documents.

Storage areas for reagents, solvents, standards, and reference materials should be adequate to preserve their identity, concentration, purity, and stability.

Adequate facilities should be provided for the collection and storage of all wastes, and these facilities should be operated so as to minimize environmental contamination. Waste storage and disposal facilities should comply with applicable federal, state, and local regulations.

The location of long-term and short-term storage of laboratory records and the measures to ensure the integrity of the data should be specified.

#### 4.2 EQUIPMENT/INSTRUMENTATION

Equipment and instrumentation should meet the requirements and specifications of the specific test methods and other procedures as specified in the QAPjP. The laboratory should maintain an equipment/instrument description list that includes the manufacturer, model number, year of purchase, accessories, and any modifications, updates, or upgrades that have been made.

#### 4.3 OPERATING PROCEDURES

The QAPjP should describe or make reference to all laboratory activities that may affect data quality. For routinely performed activities, SOPs are often prepared to ensure consistency and to save time and effort in preparing QAPjPs. Any deviation from an established procedure during a data collection activity should be documented. It is recommended that procedures be available for the indicated activities, and include, at a minimum, the information described below.

#### 4.3.1 Sample Management

The procedures describing the receipt, handling, scheduling, and storage of samples should be specified.

Sample Receipt and Handling -- These procedures describe the precautions to be used in opening sample shipment containers and how to verify that chain-of-custody has been maintained, examine samples for damage, check for proper preservatives and temperature, and log samples into the laboratory sample streams.

Sample Scheduling -- These procedures describe the sample scheduling in the laboratory and includes procedures used to ensure that holding time requirements are met.

Sample Storage -- These procedures describe the storage conditions for all samples, verification and documentation of daily storage temperature, and how to ensure that custody of the samples is maintained while in the laboratory.

#### 4.3.2 Reagent/Standard Preparation

The procedures describing how to prepare standards and reagents should be specified. Information concerning specific grades of materials used in reagent and standard preparation, appropriate glassware and containers for preparation and storage, and labeling and recordkeeping for stocks and dilutions should be included.

#### 4.3.3 General Laboratory Techniques

The procedures describing all essentials of laboratory operations that are not addressed elsewhere should be specified. These techniques should include, but are not limited to, glassware cleaning procedures, operation of analytical balances, pipetting techniques, and use of volumetric glassware.

#### 4.3.4 Test Methods

Procedures for test methods describing how the analyses are actually performed in the laboratory should be specified. A simple reference to standard methods is not sufficient, unless the analysis is performed exactly as described in the published method. Whenever methods from SW-846 are not appropriate, recognized methods from source documents published by the EPA, American Public Health Association (APHA), American Society for Testing and Materials (ASTM), the National Institute for Occupational Safety and Health (NIOSH), or other recognized organizations with appropriate expertise should be used, if possible. The documentation of the actual laboratory procedures for analytical methods should include the following:

Sample Preparation and Analysis Procedures -- These include applicable holding time, extraction, digestion, or preparation steps as appropriate to the method; procedures for determining the appropriate dilution to

analyze; and any other information required to perform the analysis accurately and consistently.

Instrument Standardization -- This includes concentration(s) and frequency of analysis of calibration standards, linear range of the method, and calibration acceptance criteria.

Sample Data -- This includes recording requirements and documentation including sample identification number, analyst, data verification, date of analysis and verification, and computational method(s).

Precision and Bias -- This includes all analytes for which the method is applicable and the conditions for use of this information.

Detection and Reporting Limits -- This includes all analytes in the method.

Test-Specific QC -- This describes QC activities applicable to the specific test and references any applicable QC procedures.

#### 4.3.5 Equipment Calibration and Maintenance

The procedures describing how to ensure that laboratory equipment and instrumentation are in working order should be specified. These procedures include calibration procedures and schedules, maintenance procedures and schedules, maintenance logs, service arrangements for all equipment, and spare parts available in-house. Calibration and maintenance of laboratory equipment and instrumentation should be in accordance with manufacturers' specifications or applicable test specifications and should be documented.

#### 4.3.6 QC

The type, purpose, and frequency of QC samples to be analyzed in the laboratory and the acceptance criteria should be specified. Information should include the applicability of the QC sample to the analytical process, the statistical treatment of the data, and the responsibility of laboratory staff and management in generating and using the data. Further details on development of project-specific QC protocols are described in Section 4.4.

#### 4.3.7 Corrective Action

The procedures describing how to identify and correct deficiencies in the analytical process should be specified. These should include specific steps to take in correcting the deficiencies such as preparation of new standards and reagents, recalibration and restandardization of equipment, reanalysis of samples, or additional training of laboratory personnel in methods and procedures. The procedures should specify that each corrective action should be documented with a description of the deficiency and the corrective action taken, and should include the person(s) responsible for implementing the corrective action.

#### 4.3.8 Data Reduction and Validation

The procedures describing how to review and validate the data should be specified. They should include procedures for computing and interpreting the results from QC samples, and independent procedures to verify that the analytical results are reported correctly. In addition, routine procedures used to monitor precision and bias, including evaluations of reagent, equipment rinsate, and trip blanks, calibration standards, control samples, duplicate and matrix spike samples, and surrogate recovery, should be detailed in the procedures. More detailed validation procedures should be performed when required in the contract or QAPjP.

#### 4.3.9 Reporting

The procedures describing the process for reporting the analytical results should be specified.

#### 4.3.10 Records Management

The procedures describing the means for generating, controlling, and archiving laboratory records should be specified. The procedures should detail record generation and control, and the requirements for record retention, including type, time, security, and retrieval and disposal authorities.

Project-specific records may include correspondence, chain-of-custody records, request for analysis, calibration data records, raw and finished analytical and QC data, data reports, and procedures used.

Laboratory operations records may include laboratory notebooks, instrument performance logs and maintenance logs in bound notebooks with prenumbered pages; laboratory benchsheets; software documentation; control charts; reference material certification; personnel files; laboratory procedures; and corrective action reports.

#### 4.3.11 Waste Disposal

The procedures describing the methods for disposal of chemicals including standard and reagent solutions, process waste, and samples should be specified.

### 4.4 LABORATORY QA AND QC PROCEDURES

The QAPjP should describe how the following required elements of the laboratory QC program are to be implemented.

#### 4.4.1 Method Proficiency

Procedures should be in place for demonstrating proficiency with each analytical method routinely used in the laboratory. These should include procedures for demonstrating the precision and bias of the method as performed by the laboratory and procedures for determining the method detection limit

(MDL). All terminology, procedures and frequency of determinations associated with the laboratory's establishment of the MDL and the reporting limit should be well-defined and well-documented. Documented precision, bias, and MDL information should be maintained for all methods performed in the laboratory.

#### 4.4.2 Control Limits

Procedures should be in place for establishing and updating control limits for analysis. Control limits should be established to evaluate laboratory precision and bias based on the analysis of control samples. Typically, control limits for bias are based on the historical mean recovery plus or minus three standard deviation units, and control limits for precision range from zero (no difference between duplicate control samples) to the historical mean relative percent difference plus three standard deviation units. Procedures should be in place for monitoring historical performance and should include graphical (control charts) and/or tabular presentations of the data.

#### 4.4.3 Laboratory Control Procedures

Procedures should be in place for demonstrating that the laboratory is in control during each data collection activity. Analytical data generated with laboratory control samples that fall within prescribed limits are judged to be generated while the laboratory was in control. Data generated with laboratory control samples that fall outside the established control limits are judged to be generated during an "out-of-control" situation. These data are considered suspect and should be repeated or reported with qualifiers.

Laboratory Control Samples -- Laboratory control samples should be analyzed for each analytical method when appropriate for the method. A laboratory control sample consists of either a control matrix spiked with analytes representative of the target analytes or a certified reference material.

Laboratory control sample(s) should be analyzed with each batch of samples processed to verify that the precision and bias of the analytical process are within control limits. The results of the laboratory control sample(s) are compared to control limits established for both precision and bias to determine usability of the data.

Method Blank -- When appropriate for the method, a method blank should be analyzed with each batch of samples processed to assess contamination levels in the laboratory. Guidelines should be in place for accepting or rejecting data based on the level of contamination in the blank.

Procedures should be in place for documenting the effect of the matrix on method performance. When appropriate for the method, there should be at least one matrix spike and either one matrix duplicate or one matrix spike duplicate per analytical batch. Additional control samples may be necessary to assure data quality to meet the project-specific DQOs.

Matrix-Specific Bias -- Procedures should be in place for determining the bias of the method due to the matrix. These procedures should include preparation and analysis of matrix spikes, selection and use of surrogates for organic methods, and the method of standard additions for metal and inorganic methods. When the concentration of the analyte in the sample is greater than 0.1%, no spike is necessary.

Matrix-Specific Precision -- Procedures should be in place for determining the precision of the method for a specific matrix. These procedures should include analysis of matrix duplicates and/or matrix spike duplicates. The frequency of use of these techniques should be based on the DQO for the data collection activity.

Matrix-Specific Detection Limit -- Procedures should be in place for determining the MDL for a specific matrix type (e.g., wastewater treatment sludge, contaminated soil, etc).

#### 4.4.4 Deviations

Any activity not performed in accordance with laboratory procedures or QAPjPs is considered a deviation from plan. All deviations from plan should be documented as to the extent of, and reason for, the deviation.

#### 4.4.5 Corrective Action

Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria should be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken should be documented.

#### 4.4.6 Data Handling

Data resulting from the analyses of samples should be reduced according to protocols described in the laboratory procedures. Computer programs used for data reduction should be validated before use and verified on a regular basis. All information used in the calculations (e.g., raw data, calibration files, tuning records, results of standard additions, interference check results, and blank- or background-correction protocols) should be recorded in order to enable reconstruction of the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) should also be maintained in order to enable reconstruction of the final result at a later date.

All data should be reviewed by a second analyst or supervisor according to laboratory procedures to ensure that calculations are correct and to detect transcription errors. Spot checks should be performed on computer calculations to verify program validity. Errors detected in the review process should be referred to the analyst(s) for corrective action. Data should be reported in accordance with the requirements of the end-user. It is recommended that the supporting documentation include at a minimum:



- Laboratory name and address.
- Sample information (including unique sample identification, sample collection date and time, date of sample receipt, and date(s) of sample preparation and analysis).
- Analytical results reported with an appropriate number of significant figures.
- Detection limits that reflect dilutions, interferences, or correction for equivalent dry weight.
- Method reference.
- Appropriate QC results (correlation with sample batch should be traceable and documented).
- Data qualifiers with appropriate references and narrative on the quality of the results.

#### 4.5 QUALITY ASSURANCE REVIEW

The QA review consists of internal and external assessments to ensure that QA/QC procedures are in use and to ensure that laboratory staff conform to these procedures. QA review should be conducted as deemed appropriate and necessary.

#### 4.6 LABORATORY RECORDS

Records provide the direct evidence and support for the necessary technical interpretations, judgements, and discussions concerning project activities. These records, particularly those that are anticipated to be used as evidentiary data, should directly support technical studies and activities, and provide the historical evidence needed for later reviews and analyses. Records should be legible, identifiable, and retrievable, and protected against damage, deterioration, or loss. The discussion in this section (4.6) outlines recommended procedures for record keeping. Organizations which conduct field sampling should develop appropriate record keeping procedures which satisfy relevant technical and legal requirements.

Laboratory records generally consist of bound notebooks with prenumbered pages, personnel qualification and training forms, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and analytical change request forms. All records should be written in indelible ink.

Procedures for reviewing, approving, and revising laboratory records should be clearly defined, with the lines of authority included. Any documentation errors should be corrected by drawing a single line through the error so that it remains legible and should be initialed by the responsible individual, along with the date of change. The correction is written adjacent to the error.

Strip-chart recorder printouts should be signed by the person who performed the instrumental analysis. If corrections need to be made in computerized data, a system parallel to the corrections for handwritten data should be in place.

Records of sample management should be available to permit the re-creation of an analytical event for review in the case of an audit or investigation of a dubious result.

Laboratory records should include, at least, the following:

Operating Procedures -- Procedures should be available to those performing the task outlined. Any revisions to laboratory procedures should be written; dated, and distributed to all affected individuals to ensure implementation of changes. Areas covered by operating procedures are given in Sections 3.3 and 4.3.

Quality Assurance Plans -- The QAPjP should be on file.

Equipment Maintenance Documentation -- A history of the maintenance record of each system serves as an indication of the adequacy of maintenance schedules and parts inventory. As appropriate, the maintenance guidelines of the equipment manufacturer should be followed. When maintenance is necessary, it should be documented in either standard forms or in logbooks. Maintenance procedures should be clearly defined and written for each measurement system and required support equipment.

Proficiency -- Proficiency information on all compounds reported should be maintained and should include (1) precision; (2) bias; (3) method detection limits; (4) spike recovery, where applicable; (5) surrogate recovery, where applicable; (6) checks on reagent purity, where applicable; and (7) checks on glassware cleanliness, where applicable.

Calibration Records & Traceability of Standards/Reagents -- Calibration is a reproducible reference point to which all sample measurements can be correlated. A sound calibration program should include provisions for documenting frequency, conditions, standards, and records reflecting the calibration history of a measurement system. The accuracy of the calibration standards is important because all data will be in reference to the standards used. A program for verifying and documenting the accuracy and traceability of all working standards against appropriate primary grade standards or the highest quality standards available should be routinely followed.

Sample Management -- All required records pertaining to sample management should be maintained and updated regularly. These include chain-of-custody forms, sample receipt forms, and sample disposition records.

Original Data -- The raw data and calculated results for all samples should be maintained in laboratory notebooks, logs, benchsheets, files or other sample tracking or data entry forms. Instrumental output should be stored in a computer file or a hardcopy report.

QC Data -- The raw data and calculated results for all QC and field samples and standards should be maintained in the manner described in the preceding paragraph. Documentation should allow correlation of sample results with associated QC data. Documentation should also include the source and lot numbers of standards for traceability. QC samples include, but are not limited to, control samples, method blanks, matrix spikes, and matrix spike duplicates.

Correspondence -- Project correspondence can provide evidence supporting technical interpretations. Correspondence pertinent to the project should be kept and placed in the project files.

Deviations -- All deviations from procedural and planning documents should be recorded in laboratory notebooks. Deviations from QAPjPs should be reviewed and approved by the authorized personnel who performed the original technical review or by their designees.

Final Report -- A copy of any report issued and any supporting documentation should be retained.

## 5.0 DEFINITIONS

The following terms are defined for use in this document:

- ACCURACY**            The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.
- BATCH:**              A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit (see Section 3.4.1 for field samples and Section 4.4.3 for laboratory samples). For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.
- BIAS:**                The deviation due to matrix effects of the measured value ( $x_s - x_u$ ) from a known spiked amount. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike). Thus, the bias (B) due to matrix effects based on a matrix spike is calculated as:

$$B = (x_s - x_u) - K$$

where:

$x_s$  = measured value for spiked sample,  
 $x_u$  = measured value for unspiked sample, and  
 $K$  = known value of the spike in the sample.

Using the following equation yields the percent recovery (%R).

$$\%R = 100 (x_s - x_u) / K$$

**BLANK:** see Equipment Rinsate, Method Blank, Trip Blank.

**CONTROL SAMPLE:** A QC sample introduced into a process to monitor the performance of the system.

**DATA QUALITY OBJECTIVES (DQOs):** A statement of the overall level of uncertainty that a decision-maker is willing to accept in results derived from environmental data (see reference 2, EPA/QAMS, July 16, 1986). This is qualitatively distinct from quality measurements such as precision, bias, and detection limit.

**DATA VALIDATION:** The process of evaluating the available data against the project DQOs to make sure that the objectives are met. Data validation may be very rigorous, or cursory, depending on project DQOs. The available data reviewed will include analytical results, field QC data and lab QC data, and may also include field records.

**DUPLICATE:** see Matrix Duplicate, Field Duplicate, Matrix Spike Duplicate.

**EQUIPMENT BLANK:** see Equipment Rinsate.

**EQUIPMENT RINSATE:** A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

**ESTIMATED QUANTITATION LIMIT (EQL):** The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs in SW-846 are provided for guidance and may not always be achievable.

FIELD DUPLICATES:	Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.
LABORATORY CONTROL SAMPLE:	A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.
MATRIX:	The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.
MATRIX DUPLICATE:	An intralaboratory split sample which is used to document the precision of a method in a given sample matrix.
MATRIX SPIKE:	An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.
MATRIX SPIKE DUPLICATES:	Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.
METHOD BLANK:	<p>An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.</p> <p>For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern should not be higher than the highest of either:</p> <ul style="list-style-type: none"> <li>(1) The method detection limit, or</li> <li>(2) Five percent of the regulatory limit for that analyte, or</li> <li>(3) Five percent of the measured concentration in the sample.</li> </ul>
METHOD DETECTION LIMIT (MDL):	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from

analysis of a sample in a given matrix type containing the analyte.

For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of three analyses of a matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL, where the t-statistic is obtained from standard references or the table below.

<u>No. of samples:</u>	<u>t-statistic</u>
3	6.96
4	4.54
5	3.75
6	3.36
7	3.14
8	3.00
9	2.90
10	2.82

Estimate the MDL as follows:

Obtain the concentration value that corresponds to:

- a) an instrument signal/noise ratio within the range of 2.5 to 5.0, or
- b) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).

Determine the variance ( $S^2$ ) for each analyte as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n (x_i - \bar{x})^2 \right]$$

where  $x_i$  = the  $i$ th measurement of the variable  $x$   
and  $\bar{x}$  = the average value of  $x$ ;

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

Determine the standard deviation (s) for each analyte as follows:

$$s = (S^2)^{1/2}$$

Determine the MDL for each analyte as follows:

$$MDL = t_{(n-1, \alpha = .99)}(s)$$

where  $t_{(n-1, \alpha = .99)}$  is the one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99 percent level.

#### ORGANIC-FREE REAGENT WATER:

For volatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. Organic-free reagent water may also be prepared by boiling water for 15 minutes and, subsequently, while maintaining the temperature at 90°C, bubbling a contaminant-free inert gas through the water for 1 hour.

For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

#### PRECISION:

The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are the relative standard deviation (RSD) or the coefficient of variation (CV),

$$RSD = CV = 100 S/\bar{x},$$

where:

$\bar{x}$  = the arithmetic mean of the  $x_i$  measurements, and  $S$  = variance; and the relative percent difference (RPD) when only two samples are available.

$$RPD = 100 [(x_1 - x_2)/((x_1 + x_2)/2)].$$

PROJECT:	Single or multiple data collection activities that are related through the same planning sequence.
QUALITY ASSURANCE PROJECT PLAN (QAPJP):	An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.
RCRA:	The Resource Conservation and Recovery Act.
REAGENT BLANK:	See Method Blank.
REAGENT GRADE:	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
REAGENT WATER:	Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.
REFERENCE MATERIAL:	A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.
SPLIT SAMPLES:	Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples should be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra- or interlaboratory precision.
STANDARD ADDITION:	The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.
STANDARD CURVE:	A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate



section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

**SURROGATE:** An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

**TRIP BLANK:** A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

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<sup>\*</sup> Definition of term.

**APPENDIX B2**

**ORGANIC ANALYTES**

## CHAPTER FOUR

### ORGANIC ANALYTES

#### 4.1 GENERAL CONSIDERATIONS

##### 4.1.1 Introduction

Following the initial and critical step of designing a sampling plan (Chapter Nine) is the implementation of that plan such that a representative sample of the solid waste is collected. Once the sample has been collected it must be stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. The sample type; type of containers and their preparation, possible forms of contamination, and preservation methods are all items which must be thoroughly examined in order to maintain the integrity of the samples. This section highlights considerations which must be addressed in order to maintain a sample's integrity and representativeness. This section is, however, applicable only to trace analyses.

Quality Control requirements need not be met for all compounds presented in the Table of Analytes for the method in use, rather, they must be met for all compounds reported. A report of non-detect is considered a quantitative report, and must meet all applicable QC requirements for that compound and the method used.

##### 4.1.2 Sample Handling and Preservation

This section deals separately with volatile and semivolatile organics. Refer to Chapter Two and Table 4-1 of this Section for recommended sample containers, sample preservation, and sample holding times.

##### Volatile Organics

Standard 40 mL glass screw-cap VOA vials with Teflon lined silicone septa may be used for both liquid and solid matrices. The vials and septa should be washed with soap and water and rinsed with distilled deionized water. After thoroughly cleaning the vials and septa, they should be placed in an oven and dried at 100 °C for approximately one hour.

**NOTE:** Do not heat the septa for extended periods of time (i.e. more than one hour, because the silicone begins to slowly degrade at 105°C).

When collecting the samples, liquids and solids should be introduced into the vials gently to reduce agitation which might drive off volatile compounds. Liquid samples should be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. Each VOA vial should be filled until there is a meniscus over the lip of the vial. The screw-top lid with the septum (Teflon side toward the sample) should then be tightened onto the vial. After tightening the lid, the vial should be inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be recollected. Two VOA vials should be filled per sample location.

VOA vials for samples with solid or semi-solid matrices (e.g., sludges) should be completely filled as best as possible. The vials should be tapped slightly as they are filled to try and eliminate as much free air space as possible. Two vials should also be filled per sample location.

VOA vials should be filled and labeled immediately at the point at which the sample is collected. They should NOT be filled near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the samples. The two vials from each sampling location should then be sealed in separate plastic bags to prevent cross-contamination between samples, particularly if the sampled waste is suspected of containing high levels of volatile organics. (Activated carbon may also be included in the bags to prevent cross-contamination from highly contaminated samples). VOA samples may also be contaminated by diffusion of volatile organics through the septum during shipment and storage. To monitor possible contamination, a trip blank prepared from organic-free reagent water (as defined in Chapter One) should be carried throughout the sampling, storage, and shipping process.

#### Semivolatile Organics (including Pesticides, PCBs and Herbicides.)

Containers used to collect samples for the determination of semivolatile organic compounds should be soap and water washed followed by methanol (or isopropanol) rinsing (see Section 4.1.4 for specific instructions on glassware cleaning). The sample containers should be of glass or Teflon, and have screw-caps with Teflon lined septa. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank.

#### 4.1.3 Safety

Safety should always be the primary consideration in the collection of samples. A thorough understanding of the waste production process, as well as all of the potential hazards making up the waste, should be investigated whenever possible. The site should be visually evaluated just prior to sampling to determine additional safety measures. Minimum protection of gloves and safety glasses should be worn to prevent sample contact with the skin and eyes. A respirator should be worn even when working outdoors if organic vapors are present. More hazardous sampling missions may require the use of supplied air and special clothing.

#### 4.1.4 Cleaning of Glassware

In the analysis of samples containing components in the parts per billion range, the preparation of scrupulously clean glassware is mandatory. Failure to do so can lead to a myriad of problems in the interpretation of the final chromatograms due to the presence of extraneous peaks resulting from

contamination. Particular care must be taken with glassware such as Soxhlet extractors, Kuderna-Danish evaporative concentrators, sampling-train components, or any other glassware coming in contact with an extract that will be evaporated to a smaller volume. The process of concentrating the compounds of interest in this operation may similarly concentrate the contaminating substance(s), which may seriously distort the results.

The basic cleaning steps are:

1. Removal of surface residuals immediately after use;
2. Hot soak to loosen and float most particulate material;
3. Hot water rinse to flush away floated particulates;
4. Soak with an oxidizing agent to destroy traces of organic compounds;
5. Hot water rinse to flush away materials loosened by the deep penetrant soak;
6. Distilled water rinse to remove metallic deposits from the tap water;
7. Alcohol, e.g., isopropanol or methanol, rinse to flush off any final traces of organic materials and remove the water; and
8. Flushing the item immediately before use with some of the same solvent that will be used in the analysis.

Each of these eight fundamental steps will be discussed in the order in which they appear above.

1. As soon possible after glassware (i.e. beakers, pipets, flasks, or bottles) has come in contact with sample or standards, the glassware should be flushed with alcohol before it is placed in the hot detergent soak. If this is not done, the soak bath may serve to contaminate all other glassware placed therein.
2. The hot soak consists of a bath of a suitable detergent in water of 50°C or higher. The detergent, powder or liquid, should be entirely synthetic and not a fatty acid base. There are very few areas of the country where the water hardness is sufficiently low to avoid the formation of some hard-water scum resulting from the reaction between calcium and magnesium salts with a fatty acid soap. This hard-water scum or curd would have an affinity particularly for many chlorinated compounds and, being almost wholly water-insoluble, would deposit on all glassware in the bath in a thin film.

There are many suitable detergents on the wholesale and retail market. Most of the common liquid dishwashing detergents sold at retail are satisfactory but are more expensive than other comparable products sold industrially. Alconox, in powder or tablet form, is manufactured by Alconox, Inc., New York, and is marketed by a number of laboratory supply firms. Sparkleen, another powdered product, is distributed by Fisher Scientific Company.

3. No comments required.
4. The most common and highly effective oxidizing agent for removal of traces of organic compounds is the traditional chromic acid solution made up of concentrated sulfuric acid and potassium or sodium dichromate. For maximum efficiency, the soak solution should be hot (40-50°C). Safety precautions must be rigidly observed in the handling of this solution. Prescribed safety gear should include safety goggles, rubber gloves, and apron. The bench area where this operation is conducted should be covered with fluorocarbon sheeting because spattering will disintegrate any unprotected surfaces.

The potential hazards of using chromic sulfuric acid mixture are great and have been well publicized. There are now commercially available substitutes that possess the advantage of safety in handling. These are biodegradable concentrates with a claimed cleaning strength equal to the chromic acid solution. They are alkaline, equivalent to ca. 0.1 N NaOH upon dilution, and are claimed to remove dried blood, silicone greases, distillation residues, insoluble organic residues, etc. They are further claimed to remove radioactive traces and will not attack glass or exert a corrosive effect on skin or clothing. One such product is "Chem Solv 2157," manufactured by Mallinckrodt and available through laboratory supply firms. Another comparable product is "Detex," a product of Borer-Chemie, Solothurn, Switzerland.

- 5, 6, and 7. No comments required.

8. There is always a possibility that between the time of washing and the next use, the glassware could pick up some contamination from either the air or direct contact. To ensure against this, it is good practice to flush the item immediately before use with some of the same solvent that will be used in the analysis.

The drying and storage of the cleaned glassware is of critical importance to prevent the beneficial effects of the scrupulous cleaning from being nullified. Pegboard drying is not recommended. It is recommended that laboratory glassware and equipment be dried at 100 °C. Under no circumstances should such small items be left in the open without protective covering. The dust cloud raised by the daily sweeping of the laboratory floor can most effectively recontaminate the clean glassware.

As an alternate to solvent rinsing, the glassware can be heated to a minimum of 300 °C to vaporize any organics. Do not use this high temperature treatment on volumetric glassware, glassware with ground glass joints, or sintered glassware.

#### 4.1.5 High Concentration Samples

Cross contamination of trace concentration samples may occur when prepared in the same laboratory with high concentration samples. Ideally, if both type samples are being handled, a laboratory and glassware dedicated solely to the preparation of high concentration samples would be available for this purpose. If this is not feasible, as a minimum when preparing high concentration samples, disposable glassware should be used or, at least, glassware dedicated entirely to the high concentration samples. Avoid cleaning glassware used for both trace and high concentration samples in the same area.



TABLE 4-1.  
RECOMMENDED SAMPLE CONTAINERS, PRESERVATION  
TECHNIQUES, AND HOLDING TIMES

Analyte Class	Container	Preservative	Holding Time
<u>Volatile Organics</u>			
Concentrated Waste Samples	8 oz. widemouth. glass with Teflon liner	None.	14 days
Liquid Samples			
No Residual Chlorine Present	2 X 40 mL vials with Teflon lined septum caps.	Cool, 4°C. <sup>1</sup>	14 days
Residual Chlorine Present	2 X 40 mL vials with Teflon lined septum caps.	Collect sample in a 4 oz. soil VOA container which has been pre-preserved with 4 drops of 10% sodium thiosulfate. Gently mix sample and transfer to a 40 mL VOA vial. Cool to 4°C.	14 days
Acrolein and Acrylonitrile	2 X 40 mL vials with Teflon lined septum caps.	Adjust to pH 4-5, Cool, 4°C.	14 days
Soil/Sediments and Sludges	4 oz (120 mL) widemouth glass with Teflon liner, or wide mouth glass container sealed with a septum.	Cool, 4°C.	14 days

<sup>1</sup> Adjust pH <2 with H<sub>2</sub>SO<sub>4</sub>, HCl or solid NaHSO<sub>4</sub>.

TABLE 4-1.  
(Continued)

Analyte Class	Container	Preservative	Holding Time
<u>Semivolatile Organics/Organochlorine Pesticides/PCBs and Herbicides</u>			
Concentrated Waste Samples	8 oz. widemouth glass with Teflon liner	None	Samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.
Water Samples			
No Residual Chlorine Present	1-gal. or 2 x 0.5-gal. amber glass with Teflon liner	Cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Residual Chlorine Present	1-gal. or 2 x 0.5-gal. amber glass with Teflon liner	Add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C	Samples must be extracted within 7 days and extracts analyzed within 40 days following extraction.
Soil/Sediments and Sludges	8 oz. widemouth glass with Teflon liner	Cool, 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days following extraction.

## 4.2 SAMPLE PREPARATION METHODS

### 4.2.1 EXTRACTIONS AND PREPARATIONS

**APPENDIX B3**

**GAS CHROMATOGRAPHY**

## METHOD 8000A

### GAS CHROMATOGRAPHY

#### 1.0 SCOPE AND APPLICATION

1.1 Gas chromatography is a quantitative technique useful for the analysis of organic compounds capable of being volatilized without being decomposed or chemically rearranged. Gas chromatography (GC), also known as vapor phase chromatography (VPC), has two subcategories distinguished by: gas-solid chromatography (GSC), and gas-liquid chromatography (GLC) or gas-liquid partition chromatography (GLPC). This last group is the most commonly used, distinguished by type of column adsorbent or packing.

1.2 The chromatographic methods are recommended for use only by, or under the close supervision of, experienced residue analysts.

#### 2.0 SUMMARY OF METHOD

2.1 Each organic analytical method that follows provides a recommended technique for extraction, cleanup, and occasionally, derivatization of the samples to be analyzed. Before the prepared sample is introduced into the GC, a procedure for standardization must be followed to determine the recovery and the limits of detection for the analytes of interest. Following sample introduction into the GC, analysis proceeds with a comparison of sample values with standard values. Quantitative analysis is achieved through integration of peak area or measurement of peak height.

#### 3.0 INTERFERENCES

3.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device must be rinsed out between samples with water or solvent. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank or of water to check for cross contamination. For volatile samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide concentrations, it may be necessary to wash out the syringe or purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses.

#### 4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak height and/or peak areas is recommended.

4.2 Gas chromatographic columns - See the specific determinative method. Other packed or capillary (open-tubular) columns may be used if the requirements

of Section 8.6 are met.

## 5.0 REAGENTS

5.1 See the specific determinative method for the reagents needed.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

## 7.0 PROCEDURE

7.1 Extraction - Adhere to those procedures specified in the referring determinative method.

7.2 Cleanup and separation - Adhere to those procedures specified in the referring determinative method.

7.3 The recommended gas chromatographic columns and operating conditions for the instrument are specified in the referring determinative method.

### 7.4 Calibration

7.4.1 Establish gas chromatographic operating parameters equivalent to those indicated in Section 7.0 of the determinative method of interest. Prepare calibration standards using the procedures indicated in Section 5.0 of the determinative method of interest. Calibrate the chromatographic system using either the external standard technique (Section 7.4.2) or the internal standard technique (Section 7.4.3).

#### 7.4.2 External standard calibration procedure

7.4.2.1 For each analyte of interest, prepare calibration standards at a minimum of five concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. One of the external standards should be at a concentration near, but above, the method detection limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.4.2.2 Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g. 2-5  $\mu\text{L}$  injections, purge-and-trap, etc.). Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each analyte. Alternatively, for samples that are introduced into the gas chromatograph using a syringe, the ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the

percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve.

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}^*}{\text{Mass injected (in nanograms)}}$$

\* For multiresponse pesticides/PCBs, use the total area of all peaks used for quantitation.

7.4.2.3 The working calibration curve or calibration factor must be verified on each working day by the injection of one or more calibration standards. The frequency of verification is dependent on the detector. Detectors, such as the electron capture detector, that operate in the sub-nanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of calibration is necessary. The flame ionization detector is much less sensitive and requires less frequent verification. If the response for any analyte varies from the predicted response by more than  $\pm 15\%$ , a new calibration curve must be prepared for that analyte. For methods 8010, 8020, and 8030, see Table 3 in each method for calibration and quality control acceptance criteria.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where:

$R_1$  = Calibration Factor from first analysis.

$R_2$  = Calibration Factor from succeeding analyses.

#### 7.4.3 Internal standard calibration procedure

7.4.3.1 To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Due to these limitations, no internal standard applicable to all samples can be suggested.

7.4.3.2 Prepare calibration standards at a minimum of five concentrations for each analyte of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards and dilute to volume with an appropriate solvent. One of the standards should be at a concentration near, but above, the method detection limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.4.3.3 Inject each calibration standard using the same introduction technique that will be applied to the actual samples (e.g. 2 to 5  $\mu\text{L}$  injection, purge-and-trap, etc.). Tabulate the peak height or area responses against the concentration of each compound and internal standard. Calculate response factors (RF) for each compound as follows:

$$\text{RF} = (A_s C_{is}) / (A_{is} C_s)$$

where:

$A_s$  = Response for the analyte to be measured.

$A_{is}$  = Response for the internal standard.

$C_{is}$  = Concentration of the internal standard,  $\mu\text{g/L}$ .

$C_s$  = Concentration of the analyte to be measured,  $\mu\text{g/L}$ .

If the RF value over the working range is constant ( $< 20\%$  RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios,  $A_s/A_{is}$  versus RF.

7.4.3.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. The frequency of verification is dependent on the detector. Detectors, such as the electron capture detector, that operate in the sub-nanogram range are more susceptible to changes in detector response caused by GC column and sample effects. Therefore, more frequent verification of calibration is necessary. The flame ionization detector is much less sensitive and requires less frequent verification. If the response for any analyte varies from the predicted response by more than  $\pm 15\%$ , a new calibration curve must be prepared for that compound. For methods 8010, 8020, and 8030, see Table 3 in each method for calibration and quality control acceptance criteria.

## 7.5 Retention time windows

7.5.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multiresponse products (i.e. PCBs) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

7.5.2 Calculate the standard deviation of the three retention times (use any function of retention time; including absolute retention time, or relative retention time) for each single component standard. For multiresponse products, choose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.



7.5.2.1 Plus or minus three times the standard deviation of the retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multiresponse analytes (i.e. PCBs), the analyst should use the retention time window, but should primarily rely on pattern recognition.

7.5.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

7.5.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

## 7.6 Gas chromatographic analysis

7.6.1 Introduction of organic compounds into the gas chromatograph varies depending on the volatility of the compound. Volatile organics are primarily introduced by purge-and-trap (Method 5030). However, there are limited applications (in Method 5030) where direct injection is acceptable. Use of Method 3810 or 3820 as a screening technique for volatile organic analysis may be valuable with some sample matrices to prevent overloading and contamination of the GC systems. Semivolatile organics are introduced by direct injection.

7.6.2 The appropriate detector(s) is given in the specific method.

7.6.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with multi-concentration calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

7.6.4 Direct Injection - Inject 2-5  $\mu\text{L}$  of the sample extract using the solvent flush technique, if the extract is manually injected. Smaller volumes (1.0  $\mu\text{L}$ ) can be injected, and the solvent flush technique is not required, if automatic devices are employed. Record the volume injected to the nearest 0.05  $\mu\text{L}$  and the resulting peak size in area units or peak height.

7.6.5 If the responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.6.6 If peak detection is prevented by the presence of interferences, further cleanup is required.

7.6.7 Examples of chromatograms for the compounds of interest are frequently available in the referring analytical method.

7.6.8 Calibrate the system immediately prior to conducting any analyses (see Section 7.4). A mid-concentration standard must also be injected at intervals specified in the method and at the end of the analysis sequence. The calibration factor for each analyte to be quantitated, must not exceed a 15% difference when compared to the initial standard of the analysis sequence. When this criterion is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary (see Section 7.7) before recalibrating and proceeding with sample analysis. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analytes that exceeded the criterion.

7.6.9 Establish daily retention time windows for each analyte. Use the retention time for each analyte from Section 7.6.8 as the midpoint of the window for that day. The daily retention time window equals the midpoint  $\pm$  three times the standard deviation determined in Section 7.5.

7.6.9.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Normally, confirmation is required: on a second GC column, by GC/MS if concentration permits, or by other recognized confirmation techniques. Confirmation may not be necessary if the composition of the sample matrix is well established by prior analyses.

7.6.9.2 Validation of GC system qualitative performance: Use the mid-concentration standards interspersed throughout the analysis sequence (Section 7.6.8) to evaluate this criterion. If any of the standards fall outside their daily retention time window, the system is out of control. Determine the cause of the problem and correct it (see Section 7.7). All samples that were injected after the standard exceeding the criteria must be reinjected to avoid false negatives and possibly false positives.

7.7 Suggested chromatography system maintenance - Corrective measures may require any one or more of the following remedial actions.

7.7.1 Packed columns - For instruments with injection port traps, replace the demister trap, clean, and deactivate the glass injection port insert or replace with a cleaned and deactivated insert. Inspect the injection end of the column and remove any foreign material (broken glass from the rim of the column or pieces of septa). Replace the glass wool with fresh deactivated glass wool. Also, it may be necessary to remove the first few millimeters of the packing material if any discoloration is noted, also swab out the inside walls of the column if any residue is noted. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body (described in Section 7.7.3) and/or repack/replace the column.

7.7.2 Capillary columns - Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert. Break off the first few inches, up to one foot, of the injection port side of the column. Remove the column and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the column.

7.7.3 Metal injector body - Turn off the oven and remove the analytical column when the oven has cooled. Remove the glass injection port insert (instruments with off-column injection or Grob). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.7.3.1 Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene; catching the rinsate in the beaker.

7.7.3.2 Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol, acetone, and hexane. Reassemble the injector and replace the GC column.

## 7.8 Calculations

7.8.1 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor determined in Section 7.4.2. The concentration of a specific analyte is calculated as follows:

### Aqueous samples

$$\text{Concentration } (\mu\text{g/L}) = [(A_x)(A)(V_t)(D)] / [(A_s)(V_i)(V_s)]$$

where:

- $A_x$  = Response for the analyte in the sample, units may be in area counts or peak height.
- $A$  = Amount of standard injected or purged, ng.
- $A_s$  = Response for the external standard, units same as for  $A_x$ .
- $V_i$  = Volume of extract injected,  $\mu\text{L}$ . For purge-and-trap analysis,  $V_i$  is not applicable and therefore = 1.
- $D$  = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made,  $D = 1$ , dimensionless.

$V_t$  = Volume of total extract,  $\mu\text{L}$ . For purge-and-trap analysis,  $V_t$  is not applicable and therefore = 1.

$V_s$  = Volume of sample extracted or purged, mL.

#### Nonaqueous samples

$$\text{Concentration } (\mu\text{g/kg}) = [(A_x)(A)(V_t)(D)]/[(A_s)(V_i)(W)]$$

where:

$W$  = Weight of sample extracted or purged, g. The wet weight or dry weight may be used, depending upon the specific applications of the data.

$A_x$ ,  $A_s$ ,  $A$ ,  $V_t$ ,  $D$ , and  $V_i$  have the same definition as for aqueous samples when a solid sample is purged (e.g., low concentration soil) for volatile organic analysis or for semivolatile organic and pesticide extracts. When the nonaqueous sample is extracted for purge and trap analysis,  $V_i$  = volume of methanol extract added to reagent water for purge and trap analysis.

7.8.2 Internal standard calibration - For each analyte of interest, the concentration of that analyte in the sample is calculated as follows:

#### Aqueous samples

$$\text{Concentration } (\mu\text{g/L}) = [(A_x)(C_{is})(D)]/[(A_{is})(RF)(V_s)]$$

where:

$A_x$  = Response of the analyte being measured, units may be in area counts or peak height.

$C_{is}$  = Amount of internal standard added to extract or volume purged, ng.

$D$  = Dilution factor, if a dilution was made on the sample prior to analysis. If no dilution was made,  $D = 1$ , dimensionless.

$A_{is}$  = Response of the internal standard, units same as  $A_x$ .

$RF$  = Response factor for analyte, as determined in Section 7.4.3.3.

$V_s$  = Volume of water extracted or purged, mL.

#### Nonaqueous samples

$$\text{Concentration } (\mu\text{g/kg}) = [(A_s)(C_{is})(D)]/[(A_{is})(RF)(W_s)]$$

where:

$W_s$  = Weight of sample extracted, g. Either a dry weight or wet weight may be used, depending upon the specific application of the data.

$A_s$ ,  $C_{is}$ ,  $D$ ,  $A_{is}$ , and  $RF$  have the same definition as for aqueous samples.

## 8.0 QUALITY CONTROL

8.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory should maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard should be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, an organic-free reagent water blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

8.3 For each analytical batch (up to 20 samples), a reagent blank, matrix spike, and duplicate or matrix spike duplicate should be analyzed (the frequency of the spikes may be different for different monitoring programs). The blank and spiked samples should be carried through all stages of the sample preparation and measurement steps.

8.4 The experience of the analyst performing gas chromatography is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration sample should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system should take place.

### 8.5 Required instrument QC

8.5.1 Step 7.4 requires that the %RSD vary by < 20% when comparing calibration factors to determine if a five point calibration curve is linear.

8.5.2 Section 7.4 sets a limit of  $\pm 15\%$  difference when comparing daily response of a given analyte versus the initial response. For Methods 8010, 8020, and 8030, follow the guidance on limits specified in Section 7.4.3.4. If the limit is exceeded, a new standard curve should be prepared unless instrument maintenance corrects the problem for that particular analyte.

8.5.3 Step 7.5 requires the establishment of retention time windows.

8.5.4 Section 7.6.8 sets a limit of  $\pm 15\%$  difference when comparing the response from the continuing calibration standard of a given analyte versus any succeeding standards analyzed during an analysis sequence.

8.5.5 Step 7.6.9.2 requires that all succeeding standards in an analysis sequence should fall within the daily retention time window established by the first standard of the sequence.

8.6 To establish the ability to generate acceptable accuracy and precision, the analyst should perform the following operations.

8.6.1 A quality control (QC) check sample concentrate is required containing each analyte of interest. The QC check sample concentrate may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the QC check sample concentrate should be made using stock standards prepared independently from those used for calibration.

8.6.1.1 The concentration of the QC check sample concentrate is highly dependent upon the analytes being investigated. Therefore, refer to Method 3500, Section 8.0 for the required concentration of the QC check sample concentrate.

8.6.2 Preparation of QC check samples

8.6.2.1 Volatile organic analytes (Methods 8010, 8020, and 8030) - The QC check sample is prepared by adding 200  $\mu\text{L}$  of the QC check sample concentrate (Step 8.6.1) to 100 mL of water.

8.6.2.2 Semivolatile organic analytes (Methods 8040, 8060, 8070, 8080, 8090, 8100, 8110, and 8120) - The QC check sample is prepared by adding 1.0 mL of the QC check sample concentrate (Step 8.6.1) to each of four 1-L aliquots of water.

8.6.3 Four aliquots of the well-mixed QC check sample are analyzed by the same procedures used to analyze actual samples (Section 7.0 of each of the methods). For volatile organics, the preparation/analysis process is purge-and-trap/gas chromatography. For semivolatile organics, the QC check samples should undergo solvent extraction (see Method 3500) prior to chromatographic analysis.

8.6.4 Calculate the average recovery ( $\bar{x}$ ) in  $\mu\text{g/L}$ , and the standard deviation of the recovery ( $s$ ) in  $\mu\text{g/L}$ , for each analyte of interest using the four results.

8.6.5 For each analyte compare  $s$  and  $\bar{x}$  with the corresponding acceptance criteria for precision and accuracy, respectively, given the QC Acceptance Criteria Table at the end of each of the determinative methods. If  $s$  and  $\bar{x}$  for all analytes of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual  $s$  exceeds the precision limit or any individual  $\bar{x}$  falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in each of the QC Acceptance Criteria Tables present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined.

8.6.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst should proceed according to Step 8.6.6.1 or 8.6.6.2.

8.6.6.1 Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with Step 8.6.2.

8.6.6.2 Beginning with Step 8.6.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Step 8.6.2.

8.7 The laboratory should, on an ongoing basis, analyze a reagent blank and a matrix spiked duplicate for each analytical batch (up to a maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of spiked duplicates. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.7.1 The concentration of the spike in the sample should be determined as follows:

8.7.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit, or 1 to 5 times higher than the background concentration determined in Step 8.7.2, whichever concentration would be larger.

8.7.1.2 If the concentration of a specific analyte in a water sample is not being checked against a limit specific to that analyte, the spike should be at the same concentration as the QC reference sample (Step 8.6.2) or 1 to 5 times higher than the background concentration determined in Step 8.7.2, whichever concentration would be larger. For other matrices, the recommended spiking concentration is 20 times the EQL.

8.7.1.3 For semivolatile organics, it may not be possible to determine the background concentration levels prior to spiking (e.g. maximum holding times will be exceeded). If this is the case, the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the QC reference sample concentration (Step 8.6.2). For other matrices, the recommended spiking concentration is 20 times the EQL.

8.7.2 Analyze one unspiked and one spiked sample aliquot to determine percent recovery of each of the spiked compounds.

8.7.2.1 Volatile organics - Analyze one 5-mL sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Step 8.6.1) appropriate for the background concentration in the sample. Spike a second 5-mL sample aliquot with 10  $\mu$ L of the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as  $100(A - B)/T$ , where T is the known true value of the spike.

8.7.2.2 Semivolatile organics - Analyze one sample aliquot (extract of 1-L sample) to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Step 8.6.1) appropriate for the background concentration in the sample. Spike a second 1-L sample aliquot with 1.0 mL of the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as  $100(A - B)/T$ , where T is the known true value of the spike.

8.7.3 Compare the percent recovery (p) for each analyte in a water sample with the corresponding criteria presented in the QC Acceptance Criteria Table found at the end of each of the determinative methods. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1. If spiking was performed at a concentration lower than the QC reference sample concentration (Step 8.6.2), the analyst should use either the QC acceptance criteria presented in the Tables, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of an analyte: (1) Calculate accuracy ( $x'$ ) using the equation found in the Method Accuracy and Precision as a Function of Concentration Table (appears at the end of each determinative method), substituting the spike concentration (T) for C; (2) calculate overall precision ( $S'$ ) using the equation in the same Table, substituting  $x'$  for  $x$ ; (3) calculate the range for recovery at the spike concentration as  $(100x'/T) \pm 2.44(100S'/T)\%$ .

8.7.4 If any individual p falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria should be



analyzed as described in Step 8.8.

8.8 If any analyte in a water sample fails the acceptance criteria for recovery in Step 8.7, a QC reference standard containing each analyte that failed should be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC reference standard will depend upon the number of analytes being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of analytes given in a method should be measured in the sample in Step 8.7, the probability that the analysis of a QC check standard will be required is high. In this case, the QC check standard should be routinely analyzed with the spiked sample.

8.8.1 Preparation of the QC check sample - For volatile organics, add 10  $\mu$ L of the QC check sample concentrate (Step 8.6.1 or 8.7.2) to 5 mL of water. For semivolatile organics, add 1.0 mL of the QC check sample concentrate (Step 8.6.1 or 8.7.2) to 1 L of water. The QC check sample needs only to contain the analytes that failed criteria in the test in Step 8.7. Prepare the QC check sample for analysis following the guidelines given in Method 3500 (e.g. purge-and-trap, extraction, etc.).

8.8.2 Analyze the QC check sample to determine the concentration measured (A) of each analyte. Calculate each percent recovery ( $p_s$ ) as  $100(A/T)\%$ , where T is the true value of the standard concentration.

8.8.3 Compare the percent recovery ( $p_s$ ) for each analyte with the corresponding QC acceptance criteria found in the appropriate Table in each of the methods. Only analytes that failed the test in Step 8.7 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem should be immediately identified and corrected. The result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.9 As part of the QC program for the laboratory, method accuracy for each matrix studied should be assessed and records should be maintained. After the analysis of five spiked samples (of the same matrix type) as in Step 8.7, calculate the average percent recovery ( $p$ ) and the standard deviation of the percent recovery ( $s_p$ ). Express the accuracy assessment as a percent recovery interval from  $p - 2s_p$  to  $p + 2s_p$ . If  $p = 90\%$  and  $s_p = 10\%$ , for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g. after each five to ten new accuracy measurements).

8.10 Calculate surrogate control limits as follows:

8.10.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

8.10.2 Calculate the average percent recovery ( $p$ ) and standard deviation of the percent recovery ( $s$ ) for each of the surrogates when

surrogate data from 25 to 30 samples for each matrix is available.

8.10.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

$$\begin{aligned}\text{Upper Control Limit (UCL)} &= p + 3s \\ \text{Lower Control Limit (LCL)} &= p - 3s\end{aligned}$$

8.10.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits in Tables A and B of Methods 8240 and 8270, respectively. The limits given in these methods are multi-laboratory performance based limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Step 8.10.3 should fall within those given in Tables A and B for these matrices.

8.10.5 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

8.10.6 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

8.11 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer should be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 9.0 METHOD PERFORMANCE

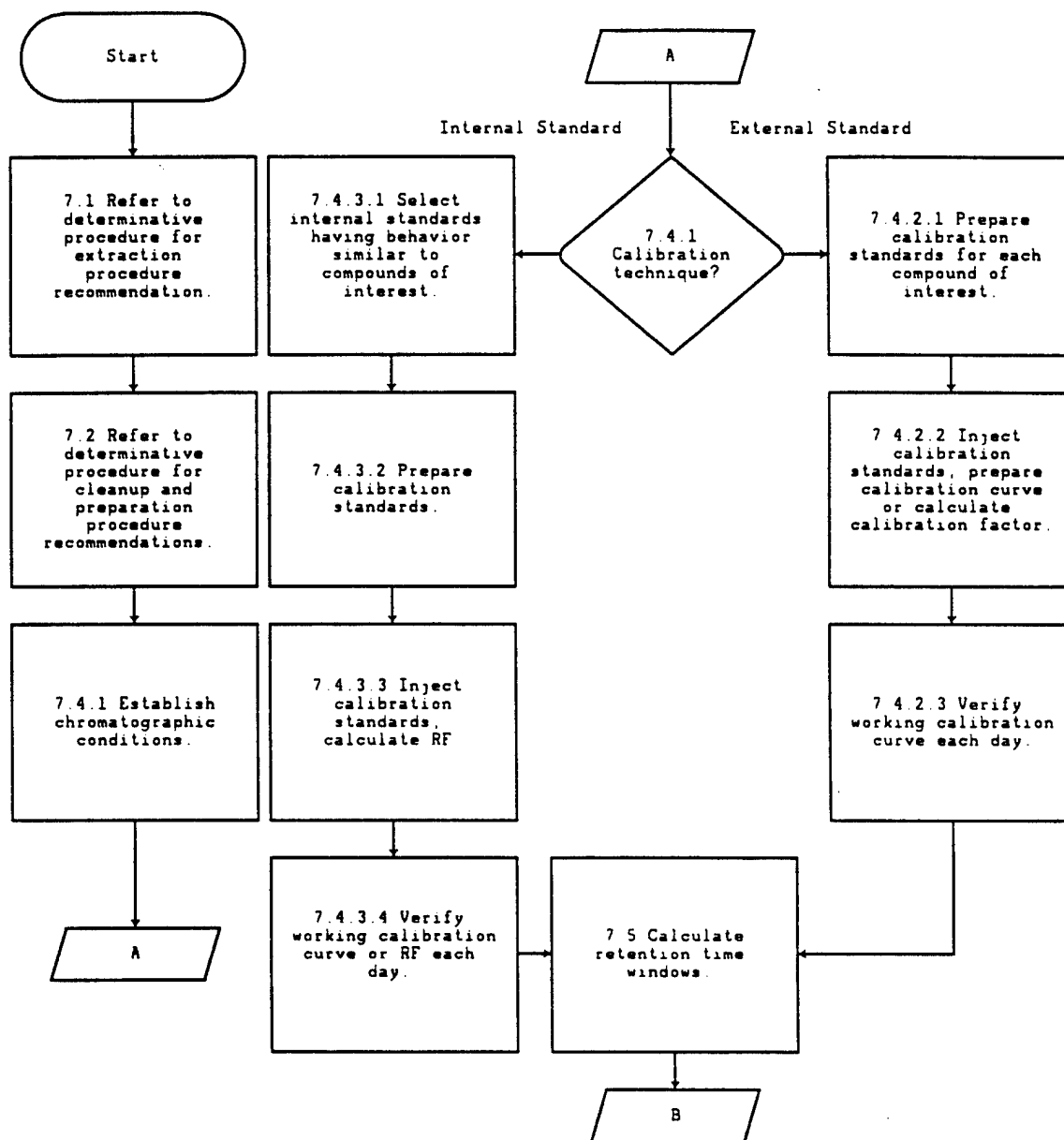
9.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in the referring analytical methods were obtained using water. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

9.2 Refer to the determinative method for specific method performance information.

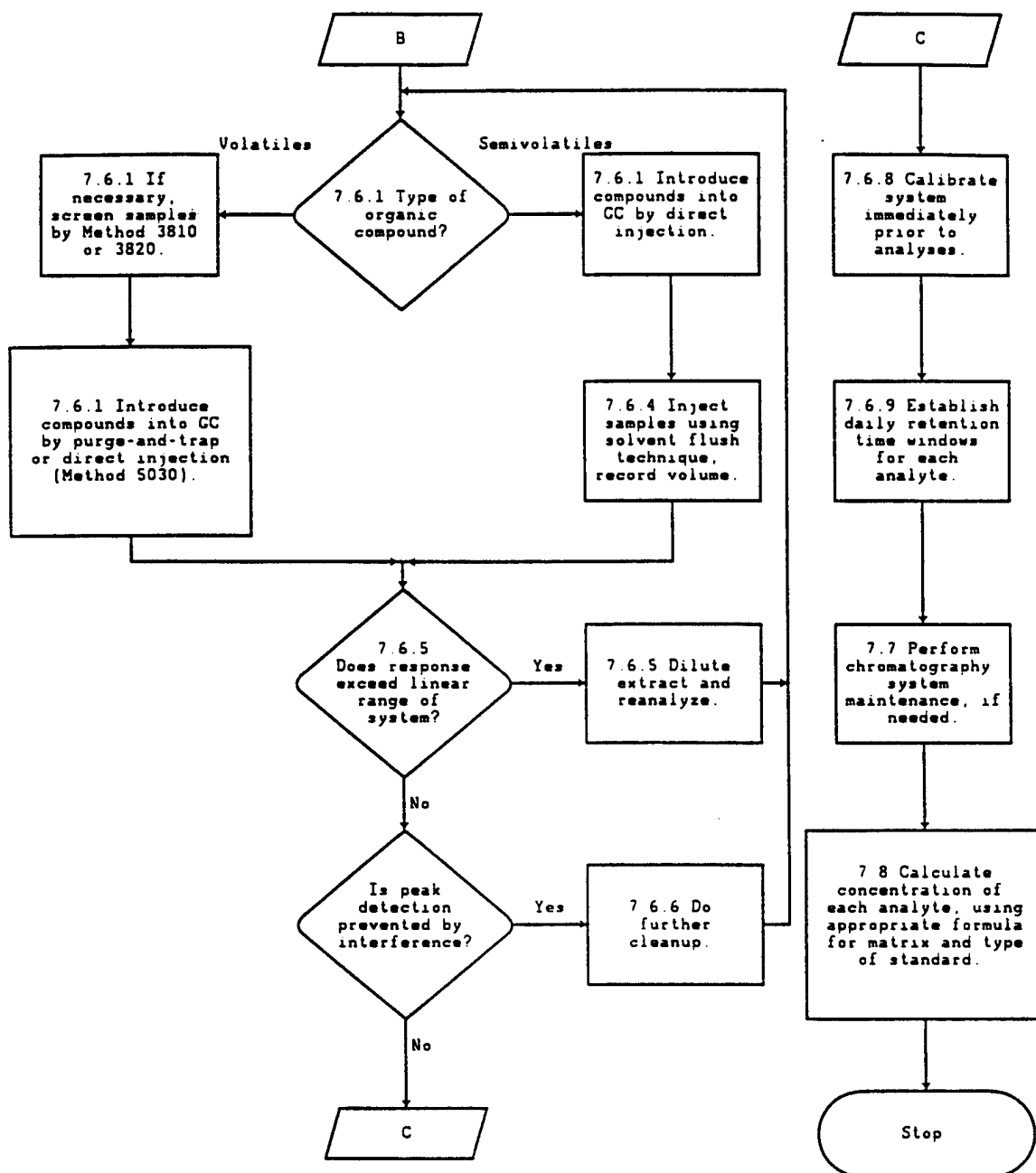
## 10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
2. U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, July 1985, Revision.

# METHOD 8000A GAS CHROMATOGRAPHY



METHOD 8000A  
continued



**APPENDIX B4**

**METHOD 8330**

**EXPLOSIVES ANALYSIS**

## METHOD 8330

### NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

#### 1.0 SCOPE AND APPLICATION

1.1 Method 8330 is intended for the trace analysis of explosives residues by high performance liquid chromatography using a UV detector. This method is used to determine the concentration of the following compounds in a water, soil, or sediment matrix:

Compound	Abbreviation	CAS No <sup>a</sup>
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	1,3-DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
2-Amino-4, 6-dinitrotoluene	2-Am-DNT	355-72-78-2
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0

a Chemical Abstracts Service Registry number

1.2 Method 8330 provides a salting-out extraction procedure for low concentration (parts per trillion or nanograms per liter) of explosives residues in surface or ground water. Direct injection of diluted and filtered water samples can be used for water samples of higher concentration (See Table 1).

1.3 All of these compounds are either used in the manufacture of explosives or are the degradation products of compounds used for that purpose. When making stock solutions for calibration, treat each explosive compound with caution. See NOTE in Section 5.3.1 and Section 11 on Safety.

1.4 The estimated quantitation limits (EQLs) of target analytes determined by Method 8330 in water and soil are presented in Table 1.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling explosive materials. (See Section 11.0 on SAFETY.) Each analyst must demonstrate the ability to generate acceptable results with this method.

## 2.0 SUMMARY OF METHOD

2.1 Method 8330 provides high performance liquid chromatographic (HPLC) conditions for the detection of ppb levels of certain explosives residues in water, soil and sediment matrix. Prior to use of this method, appropriate sample preparation techniques must be used.

2.2 Low-Level Salting-out Method With No Evaporation: Aqueous samples of low concentration are extracted by a salting-out extraction procedure with acetonitrile and sodium chloride. The small volume of acetonitrile that remains undissolved above the salt water is drawn off and transferred to a smaller volumetric flask. It is back-extracted by vigorous stirring with a specific volume of salt water. After equilibration, the phases are allowed to separate and the small volume of acetonitrile residing in the narrow neck of the volumetric flask is removed using a Pasteur pipet. The concentrated extract is diluted 1:1 with reagent grade water. An aliquot is separated on a C-18 reverse phase column, determined at 254 nm, and confirmed on a CN reverse phase column.

2.3 High-level Direct Injection Method: Aqueous samples of higher concentration can be diluted 1/1 (v/v) with methanol or acetonitrile, filtered, separated on a C-18 reverse phase column, determine at 254 nm, and confirmed on a CN reverse phase column. If HMX is an important target analyte, methanol is preferred.

2.4 Soil and sediment samples are extracted using acetonitrile in an ultrasonic bath, filtered and chromatographed as in Section 2.3.

## 3.0 INTERFERENCES

3.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

3.2 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.

3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration. All samples expected to contain tetryl should not be exposed to temperatures above room temperature.

3.4 Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.



## 4.0 APPARATUS AND MATERIALS

### 4.1 HPLC system

4.1.1 HPLC - equipped with a pump capable of achieving 4000 psi, a 100  $\mu$ l loop injector and a 254 nm UV detector (Perkin Elmer Series 3, or equivalent). For the low concentration option, the detector must be capable of a stable baseline at 0.001 absorbance units full scale.

#### 4.1.2 Recommended Columns:

4.1.2.1 Primary column: C-18 Reverse phase HPLC column, 25 cm x 4.6 mm (5  $\mu$ m), (Supelco LC-18, or equivalent).

4.1.2.2 Secondary column: CN Reverse phase HPLC column, 25 cm x 4.6 mm (5  $\mu$ m), (Supelco LC-CN, or equivalent).

#### 4.1.3 Strip chart recorder.

#### 4.1.4 Digital integrator (optional).

#### 4.1.5 Autosampler (optional).

### 4.2 Other Equipment

#### 4.2.1 Temperature controlled ultrasonic bath.

#### 4.2.2 Vortex mixer.

#### 4.2.3 Balance $\pm$ 0.0001 g.

#### 4.2.4 Magnetic stirrer with stirring pellets.

4.2.5 Water bath - Heated, with concentric ring cover, capable of temperature control ( $\pm$  5°C). The bath should be used in a hood.

#### 4.2.6 Oven - Forced air, without heating.

### 4.3 Materials

4.3.1 High pressure injection syringe - 500  $\mu$ L, (Hamilton liquid syringe or equivalent).

#### 4.3.2 Disposable cartridge filters - 0.45 $\mu$ m Teflon filter.

#### 4.3.3 Pipets - Class A, glass, Appropriate sizes.

#### 4.3.4 Pasteur pipets.

#### 4.3.5 Scintillation Vials - 20 mL, glass.

#### 4.3.6 Vials - 15 mL, glass, Teflon-lined cap.

4.3.7 Vials- 40 mL, glass, Teflon-lined cap.

4.3.8 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.

4.3.9 Volumetric flasks - Appropriate sizes with ground glass stoppers, Class A.

**NOTE:** The 100 mL and 1 L volumetric flasks used for magnetic stirrer extraction must be round.

4.3.10 Vacuum desiccator - Glass.

4.3.11 Mortar and pestle - Steel.

4.3.12 Sieve - 30 mesh.

4.3.13 Graduated cylinders - Appropriate sizes.

#### 4.4 Preparation of Materials

4.4.1 Prepare all materials to be used as described in Chapter 4 for semivolatile organics.

### 5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

5.1.1 Acetonitrile,  $\text{CH}_3\text{CN}$  - HPLC grade.

5.1.2 Methanol,  $\text{CH}_3\text{OH}$  - HPLC grade.

5.1.3 Calcium chloride,  $\text{CaCl}_2$  - Reagent grade. Prepare an aqueous solution of 5 g/L.

5.1.4 Sodium chloride,  $\text{NaCl}$ , shipped in glass bottles - reagent grade.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

#### 5.3 Stock Standard Solutions

5.3.1 Dry each solid analyte standard to constant weight in a vacuum desiccator in the dark. Place about 0.100 g (weighed to 0.0001 g) of a single analyte into a 100 mL volumetric flask and dilute to volume with acetonitrile. Invert flask several times until dissolved. Store in refrigerator at 4°C in the dark. Calculate the concentration of the stock

solution from the actual weight used (nominal concentration = 1,000 mg/L). Stock solutions may be used for up to one year.

NOTE: The HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material should be handled carefully. See SAFETY in Section 11 for guidance. HMX, RDX, and Tetryl reference materials are shipped under water. Drying at ambient temperature requires several days. DO NOT DRY AT HEATED TEMPERATURES!

#### 5.4 Intermediate Standards Solutions

5.4.1 If both 2,4-DNT and 2,6-DNT are to be determined, prepare two separate intermediate stock solutions containing (1) HMX, RDX, 1,3,5-TNB, 1,3-DNB, NB, 2,4,6-TNT, and 2,4-DNT and (2) Tetryl, 2,6-DNT, 2-NT, 3-NT, and 4-NT. Intermediate stock standard solutions should be prepared at 1,000 µg/L, in acetonitrile when analyzing soil samples, and in methanol when analyzing aqueous samples.

5.4.2 Dilute the two concentrated intermediate stock solutions, with the appropriate solvent, to prepare intermediate standard solutions that cover the range of 2.5 - 1,000 µg/L. These solutions should be refrigerated on preparation, and may be used for 30 days.

5.4.3 For the low-level method, the analyst must conduct a detection limit study and devise dilution series appropriate to the desired range. Standards for the low level method must be prepared immediately prior to use.

#### 5.5 Working standards

5.5.1 Calibration standards at a minimum of five concentration levels should be prepared through dilution of the intermediate standards solutions by 50% (v/v) with 5 g/L calcium chloride solution (Section 5.1.3). These solutions must be refrigerated and stored in the dark, and prepared fresh on the day of calibration.

#### 5.6 Surrogate Spiking Solution

5.6.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with one or two surrogates (e.g., analytes not expected to be present in the sample).

#### 5.7 Matrix Spiking Solutions

5.7.1 Prepare matrix spiking solutions in methanol such that the concentration in the sample is five times the Estimated Quantitation Limit (Table 1). All target analytes should be included.

## 5.8 HPLC Mobile Phase

5.8.1 To prepare 1 liter of mobile phase, add 500 mL of methanol to 500 mL of organic-free reagent water.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Follow conventional sampling and sample handling procedures as specified for semivolatile organics in Chapt. 4.

6.2 Samples and sample extracts must be stored in the dark at 4°C. Holding times are the same as for semivolatile organics.

## 7.0 PROCEDURE

### 7.1 Sample Preparation

7.1.1 Aqueous Samples: It is highly recommended that process waste samples be screened with the high-level method to determine if the low level method (1-50 µg/L) is required. Most groundwater samples will fall into the low level method.

#### 7.1.1.1 Low-Level Method (salting-out extraction)

7.1.1.1.1 Add 251.3 g of sodium sulfate to a 1 L volumetric flask (round). Measure out 770 mL of a water sample (using a 1 L graduated cylinder) and transfer it to the volumetric flask containing the salt. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

7.1.1.1.2 Add 164 mL of acetonitrile (measured with a 250 mL graduated cylinder) while the solution is being stirred and stir for an additional 15 minutes. Turn off the stirrer and allow the phases to separate for 10 minutes.

7.1.1.1.3 Remove the acetonitrile (upper) layer (about 8 mL) with a Pasteur pipet and transfer it to a 100 mL volumetric flask (round). Add 10 mL of fresh acetonitrile to the water sample in the 1 L flask. Again stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract. The inclusion of a few drops of salt water at this point is unimportant.

7.1.1.1.4 Add 84 mL of salt water (325 g NaCl per 1000 mL of reagent water) to the acetonitrile extract in the 100 mL volumetric flask. Add a stir bar and stir the contents on a magnetic stirrer for 15 minutes followed by 10 minutes for phase separation. Carefully transfer the acetonitrile phase to a 10 mL graduated cylinder using a Pasteur pipet. At this

stage the amount of water transferred with the acetonitrile must be minimized. The water contains a high concentration of NaCl that produces a large peak at the beginning of the chromatogram where it could interfere with the HMX determination.

7.1.1.1.5 Add an additional 1.0 mL of acetonitrile to the 100 mL volumetric flask. Again stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 10 mL graduated cylinder (transfer to a 25 mL graduated cylinder if the volume exceeds 5 mL). Record the total volume of acetonitrile extract to the nearest 0.1 mL. (Use this as the volume of total extract [V<sub>t</sub>] in the calculation of concentration after converting to  $\mu\text{L}$ ). The resulting extract, about 5 - 6 mL, is then diluted 1:1 with reagent water prior to analysis.

7.1.1.1.6 If the diluted extract is turbid, filter it through a 0.45 -  $\mu\text{m}$  Teflon filter using a disposable syringe. Discard the first 0.5 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4.

#### 7.1.1.2 High-Level Method

7.1.1.2.1 Sample filtration: Place a 5 mL aliquot of each water sample in a scintillation vial, add 5 mL of acetonitrile, shake thoroughly, and filter through a 0.45- $\mu\text{m}$  Teflon filter using a disposable syringe. Discard the first 3 mL of filtrate, and retain the remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration.

#### 7.1.2 Soil and Sediment Samples

7.1.2.1 Sample homogenization: Dry soil samples in air at room temperature or colder to a constant weight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile rinsed mortar to pass a 30 mesh sieve.

NOTE: Soil samples should be screened by Method 8510 prior to grinding in a mortar and pestle (See Safety Section 11.2).

#### 7.1.2.2 Sample extraction

7.1.2.2.1 Place a 2.0 g subsample of each soil sample in a 15 mL glass vial. Add 10.0 mL of acetonitrile, cap with Teflon-lined cap, vortex swirl for one minute, and place in a cooled ultrasonic bath for 18 hours.

7.1.2.2.2 After sonication, allow sample to settle for 30 minutes. Remove 5.0 mL of supernatant, and combine with 5.0 mL of calcium chloride solution (Section 5.1.3) in a 20 mL vial. Shake, and let stand for 15 minutes.

7.1.2.2.3 Place supernatant in a disposable syringe and filter through a 0.45- $\mu$ m Teflon filter. Discard first 3 mL and retain remainder in a Teflon-capped vial for RP-HPLC analysis as in Section 7.4.

## 7.2 Chromatographic Conditions (Recommended)

Primary Column: C-18 reverse phase HPLC column, 25-cm x 4.6-mm, 5  $\mu$ m, (Supelco LC-18 or equivalent).

Secondary Column: CN reverse phase HPLC column, 25-cm x 4.6-mm, 5  $\mu$ m, (Supelco LC-CN or equivalent).

Mobile Phase: 50/50 (v/v) methanol/organic-free reagent water.

Flow Rate: 1.5 mL/min

Injection volume: 100- $\mu$ L

UV Detector: 254 nm

## 7.3 Calibration of HPLC

7.3.1 All electronic equipment is allowed to warm up for 30 minutes. During this period, at least 15 void volumes of mobile phase are passed through the column (approximately 20 min at 1.5 mL/min) and continued until the baseline is level at the UV detector's greatest sensitivity.

7.3.2 Initial Calibration. Triplicate injections of each calibration standard over the concentration range of interest are sequentially injected into the HPLC in random order. Peak heights or peak areas are obtained for each analyte. Experience indicates that a linear calibration curve with zero intercept is appropriate for each analyte. Therefore, a response factor for each analyte can be taken as the slope of the best-fit regression line.

7.3.3 Daily Calibration. Analyze midpoint calibration standards, at a minimum, in triplicate at the beginning of the day, singly at the midpoint of the run and singly after the last sample of the day (assuming a sample group of 10 samples or less). Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within  $\pm 15\%$  of the response factor of the initial calibration. The same criteria is required

for subsequent standard responses compared to the mean response of the triplicate standards beginning the day. If this criterion is not met, a new initial calibration must be obtained.

#### 7.4 HPLC Analysis

7.4.1 Analyze the samples using the chromatographic conditions given in Section 7.2. All positive measurements observed on the C-18 column must be confirmed by injection onto the CN column.

7.4.2 Follow Section 7.0 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence. If column temperature control is not employed, special care must be taken to ensure that temperature shifts do not cause peak misidentification.

7.4.3 Table 2 summarizes the estimated retention times on both C-18 and CN columns for a number of analytes analyzable using this method. An example of the separation achieved by Column 1 is shown in Figure 1.

7.4.4 Record the resulting peak sizes in peak heights or area units. The use of peak heights is recommended to improve reproducibility of low level samples.

7.4.5 Calculation of concentration is covered in Section 7.0 of Method 8000.

#### 8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500.

8.2 Quality control required to validate the HPLC system operation is found in Method 8000, Section 8.0.

8.3 Prior to preparation of stock solutions, acetonitrile, methanol, and water blanks should be run to determine possible interferences with analyte peaks. If the acetonitrile, methanol, or water blanks show contamination, a different batch should be used.

#### 9.0 METHOD PERFORMANCE

9.1 Table 3 presents the single laboratory precision based on data from the analysis of blind duplicates of four spiked soil samples and four field contaminated samples analyzed by seven laboratories.

9.2 Table 4 presents the multilaboratory error based on data from the analysis of blind duplicates of four spiked soil samples and four field contaminated samples analyzed by seven laboratories.

9.3 Table 5 presents the multilaboratory variance of the high concentration method for water based on data from nine laboratories.

9.4 Table 6 presents multilaboratory recovery data from the analysis of spiked soil samples by seven laboratories.

9.5 Table 7 presents a comparison of method accuracy for soil and aqueous samples (high concentration method).

9.6 Table 8 contains precision and accuracy data for the salting-out extraction method.

## 10.0 REFERENCES

1. Bauer, C.F., T.F. Jenkins, S.M. Koza, P.W. Schumacher, P.H. Miyares and M.E. Walsh (1989). Development of an analytical method for the determination of explosive residues in soil. Part 3. Collaborative test results and final performance evaluation. USA Cold Regions Research and Engineering Laboratory, CRREL Report 89-9.
2. Grant, C.L., A.D. Hewitt and T.F. Jenkins (1989) Comparison of low concentration measurement capability estimates in trace analysis: Method Detection Limits and Certified Reporting Limits. USA Cold Regions Research and Engineering Laboratory, Special Report 89-20.
3. Jenkins, T.F., C.F. Bauer, D.C. Leggett and C.L. Grant (1984) Reversed-phased HPLC method for analysis of TNT, RDX, HMX and 2,4-DNT in munitions wastewater. USA Cold Regions Research and Engineering Laboratory, CRREL Report 84-29.
4. Jenkins, T.F. and M.E. Walsh (1987) Development of an analytical method for explosive residues in soil. USA Cold Regions Research and Engineering Laboratory, CRREL Report 87-7.
5. Jenkins, T.F., P.H. Miyares and M.E. Walsh (1988a) An improved RP-HPLC method for determining nitroaromatics and nitramines in water. USA Cold Regions Research and Engineering Laboratory, Special Report 88-23.
6. Jenkins, T.F. and P.H. Miyares (1992) Comparison of Cartridge and Membrane Solid-Phase Extraction with Salting-out Solvent Extraction for Preconcentration of Nitroaromatic and Nitramine Explosives from Water. USA Cold Regions Research and Engineering Laboratory, Draft CRREL Special Report.
7. Jenkins, T.F., P.W. Schumacher, M.E. Walsh and C.F. Bauer (1988b) Development of an analytical method for the determination of explosive residues in soil. Part II: Further development and ruggedness testing. USA Cold Regions Research and Engineering Laboratory, CRREL Report 88-8.
8. Leggett, D.C., T.F. Jenkins and P.H. Miyares (1990) Salting-out solvent extraction for preconcentration of neutral polar organic solutes from water. Analytical Chemistry, 62: 1355-1356.



9. Miyares, P.H. and T.F. Jenkins (1990) Salting-out solvent extraction for determining low levels of nitroaromatics and nitramines in water. USA Cold Regions Research and Engineering Laboratory, Special Report 90-30.

## 11.0 SAFETY

11.1 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by Method 8330. The only extra caution that should be taken is when handling the analytical standard neat material for the explosives themselves and in rare cases where soil or waste samples are highly contaminated with the explosives. Follow the note for drying the neat materials at ambient temperatures.

11.2 It is advisable to screen soil or waste samples using Method 8510 to determine whether high concentrations of explosives are present. Soil samples as high as 2% 2,4,6-TNT have been safely ground. Samples containing higher concentrations should not be ground in the mortar and pestle. Method 8510 is for 2,4,6-TNT, however, the other nitroaromatics will also cause a color to be developed and provide a rough estimation of their concentrations. 2,4,6-TNT is the analyte most often detected in high concentrations in soil samples. Visual observation of a soil sample is also important when taken from a site expected to contain explosives. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

TABLE 1  
ESTIMATED QUANTITATION LIMITS

Compounds	Water ( $\mu\text{g/L}$ )		Soil (mg/kg)
	Low-Level	High-Level	
HMX	-	13.0	2.2
RDX	0.84	14.0	1.0
1,3,5-TNB	0.26	7.3	0.25
1,3-DNB	0.11	4.0	0.25
Tetryl	-	4.0	0.65
NB	-	6.4	0.26
2,4,6-TNT	0.11	6.9	0.25
4-Am-DNT	0.060	-	-
2-Am-DNT	0.035	-	-
2,6-DNT	0.31	9.4	0.26
2,4-DNT	0.020	5.7	0.25
2-NT	-	12.0	0.25
4-NT	-	8.5	0.25
3-NT	-	7.9	0.25

TABLE 2  
RETENTION TIMES AND CAPACITY FACTORS ON LC-18 AND LC-CN COLUMNS

Compound	Retention time (min)		Capacity factor (k)*	
	LC-18	LC-CN	LC-18	LC-CN
HMX	2.44	8.35	0.49	2.52
RDX	3.73	6.15	1.27	1.59
1,3,5-TNB	5.11	4.05	2.12	0.71
1,3-DNB	6.16	4.18	2.76	0.76
Tetryl	6.93	7.36	3.23	2.11
NB	7.23	3.81	3.41	0.61
2,4,6-TNT	8.42	5.00	4.13	1.11
4-Am-DNT	8.88	5.10	4.41	1.15
2-Am-DNT	9.12	5.65	4.56	1.38
2,6-DNT	9.82	4.61	4.99	0.95
2,4-DNT	10.05	4.87	5.13	1.05
2-NT	12.26	4.37	6.48	0.84
4-NT	13.26	4.41	7.09	0.86
3-NT	14.23	4.45	7.68	0.88

\* Capacity factors are based on an unretained peak for nitrate at 1.71 min on LC-18 and 2.00 min on LC-CN

TABLE 3  
SINGLE LABORATORY PRECISION OF METHOD FOR SOIL SAMPLES

	<u>Spiked Soils</u>			<u>Field-Contaminated Soils</u>		
	Mean Conc. (mg/kg)	SD	%rsd	Mean Conc. (mg/kg)	SD	%rsd
HMX	46	1.7	3.7	14	1.8	12.8
				153	21.6	14.1
RDX	60	1.4	2.3	104	12	11.5
				877	29.6	3.4
1,3,5-TNB	8.6	0.4	4.6	2.8	0.2	7.1
	46	1.9	4.1	72	6.0	8.3
1,3-DNB	3.5	0.14	4.0	1.1	0.11	9.8
Tetryl	17	3.1	17.9	2.3	0.41	18.0
2,4,6-TNT	40	1.4	3.5	7.0	0.61	9.0
				669	55	8.2
2,4-DNT	5.0	0.17	3.4	1.0	0.44	42.3

TABLE 4  
MULTILABORATORY ERROR OF METHOD FOR SOIL SAMPLES

	Spiked Soils			Field-Contaminated Soils		
	Mean Conc. (mg/kg)	SD	%rsd	Mean Conc. (mg/kg)	SD	%rsd
HMX	46	2.6	5.7	14	3.7	26.0
				153	37.3	24.0
RDX	60	2.6	4.4	104	17.4	17.0
				877	67.3	7.7
1,3,5-TNB	8.6	0.61	7.1	2.8	0.23	8.2
	46	2.97	6.5	72	8.8	12.2
1,3-DNB	3.5	0.24	6.9	1.1	0.16	14.5
Tetryl	17	5.22	30.7	2.3	0.49	21.3
2,4,6-TNT	40	1.88	4.7	7.0	1.27	18.0
				669	63.4	9.5
2,4-DNT	5.0	0.22	4.4	1.0	0.74	74.0

TABLE 5  
MULTILABORATORY VARIANCE OF METHOD FOR WATER SAMPLES<sup>a</sup>

Compounds	Mean Conc. (µg/L)	SD	%rsd
HMX	203	14.8	7.3
RDX	274	20.8	7.6
2,4-DNT	107	7.7	7.2
2,4,6-TNT	107	11.1	10.4

<sup>a</sup> Nine Laboratories

TABLE 6  
MULTILABORATORY RECOVERY DATA FOR SPIKED SOIL SAMPLES

Laboratory	Concentration (µg/g)						
	HMX	RDX	1,3,5-TNB	1,3-DNB	Tetryl	2,4,6-TNT	2,4-DNT
1	44.97	48.78	48.99	49.94	32.48	49.73	51.05
3	50.25	48.50	45.85	45.96	47.91	46.25	48.37
4	42.40	44.00	43.40	49.50	31.60	53.50	50.90
5	46.50	48.40	46.90	48.80	32.10	55.80	49.60
6	56.20	55.00	41.60	46.30	13.20	56.80	45.70
7	41.50	41.50	38.00	44.50	2.60	36.00	43.50
8	52.70	52.20	48.00	48.30	44.80	51.30	49.10
true conc	50.35	50.20	50.15	50.05	50.35	50.65	50.05
mean	47.79	48.34	44.68	47.67	29.24	49.91	48.32
std dev	5.46	4.57	3.91	2.09	16.24	7.11	2.78
% rsd	11.42	9.45	8.75	4.39	55.53	14.26	5.76
% diff*	5.08	3.71	10.91	4.76	41.93	1.46	3.46
mean % recovery	95	96	89	95	58	98	96

\* Between true value and mean determined value.

TABLE 7  
COMPARISON OF METHOD ACCURACY FOR SOIL AND AQUEOUS SAMPLES  
(HIGH CONCENTRATION METHOD)

Analyte	Recovery (%)	
	Soil Method*	Aqueous Method**
2,4-DNT	96.0	98.6
2,4,6-TNT	96.8	94.4
RDX	96.8	99.6
HMX	95.4	95.5

\* Taken from Bauer et al. (1989), Reference 1.

\*\* Taken from Jenkins et al. (1984), Reference 3.

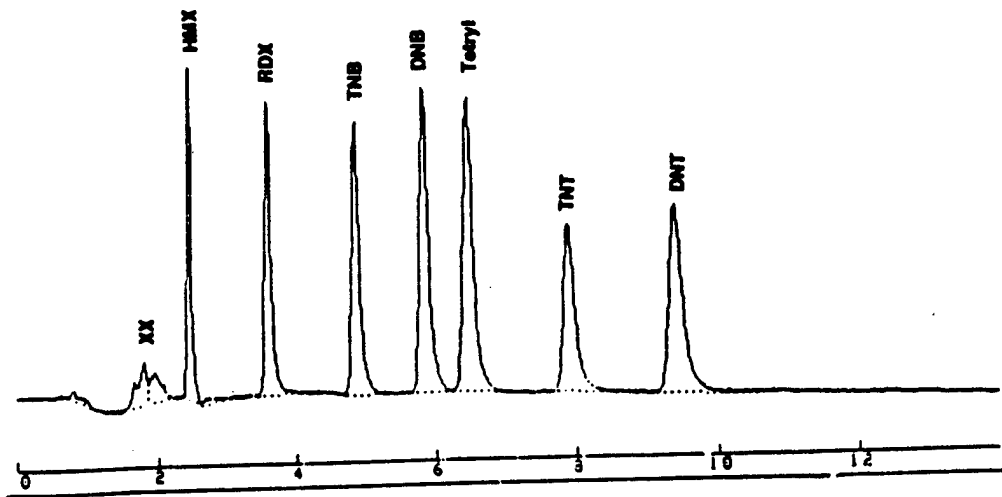
TABLE 8  
PRECISION AND ACCURACY DATA FOR THE SALTING-OUT EXTRACTION METHOD

Analyte	No. of Samples <sup>1</sup>	Precision (% RSD)	Ave. Recovery (%)	Conc. Range (µg/L)
HMX	20	10.5	106	0-1.14
RDX	20	8.7	106	0-1.04
1,3,5-TNB	20	7.6	119	0-0.82
1,3-DNB	20	6.6	102	0-1.04
Tetryl	20	16.4	93	0-0.93
2,4,6-TNT	20	7.6	105	0-0.98
2-Am-DNT	20	9.1	102	0-1.04
2,4-DNT	20	5.8	101	0-1.01
1,2-NT	20	9.1	102	0-1.07
1,4-NT	20	18.1	96	0-1.06
1,3-NT	20	12.4	97	0-1.23

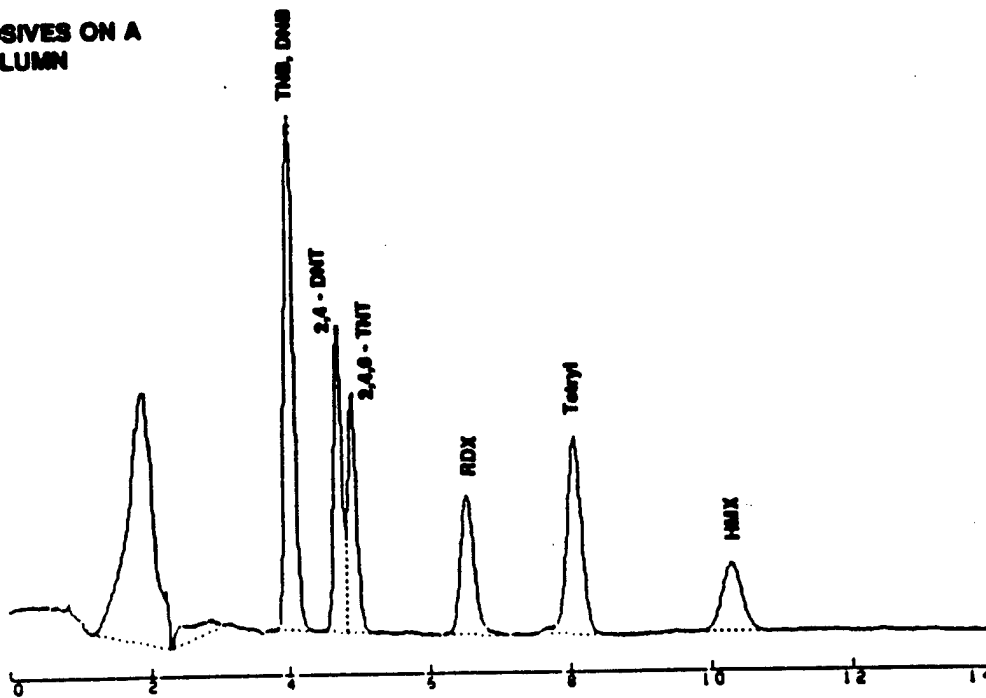
<sup>1</sup>Reagent water



**EXPLOSIVES ON A  
C18 COLUMN**

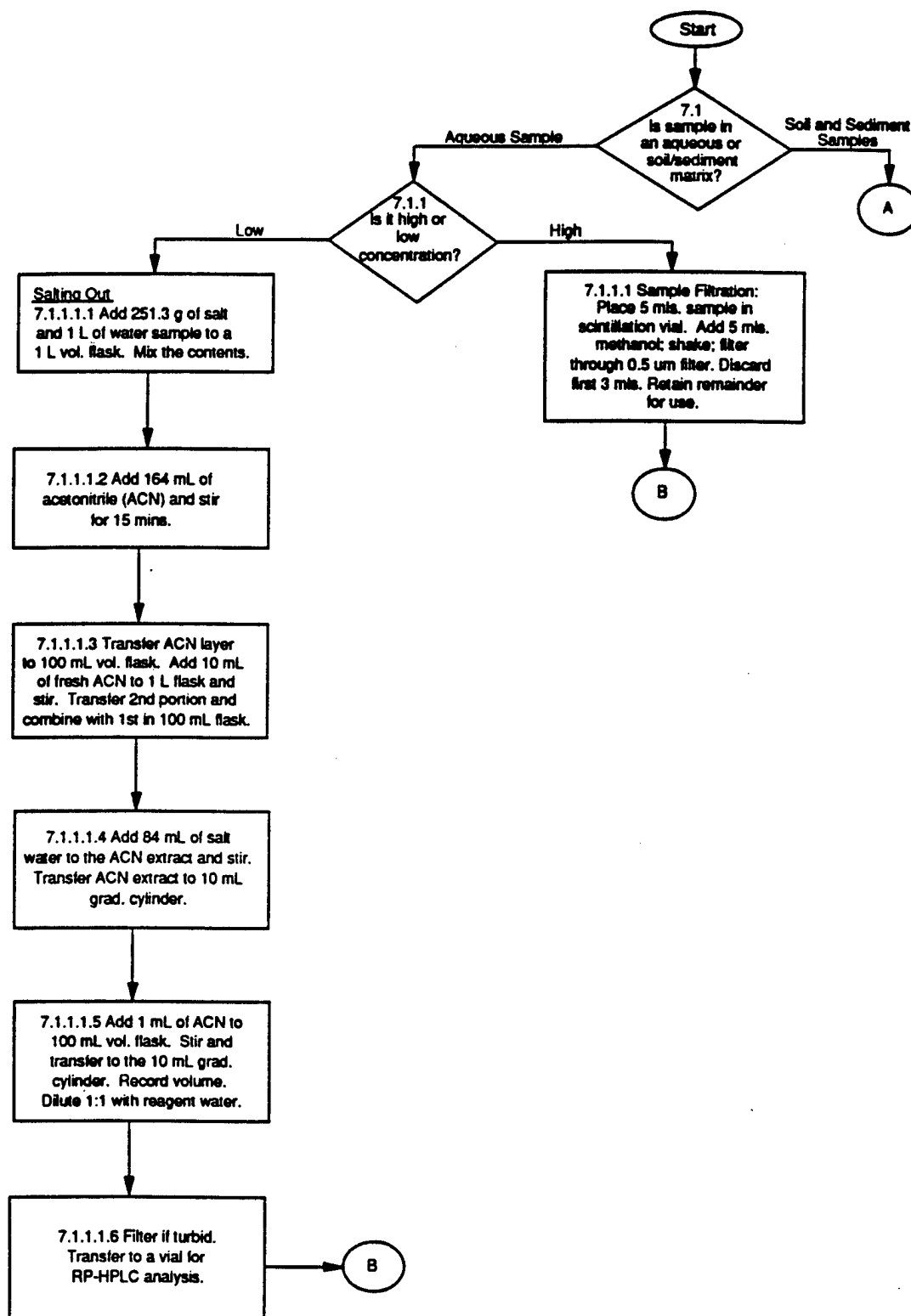


**EXPLOSIVES ON A  
CN COLUMN**

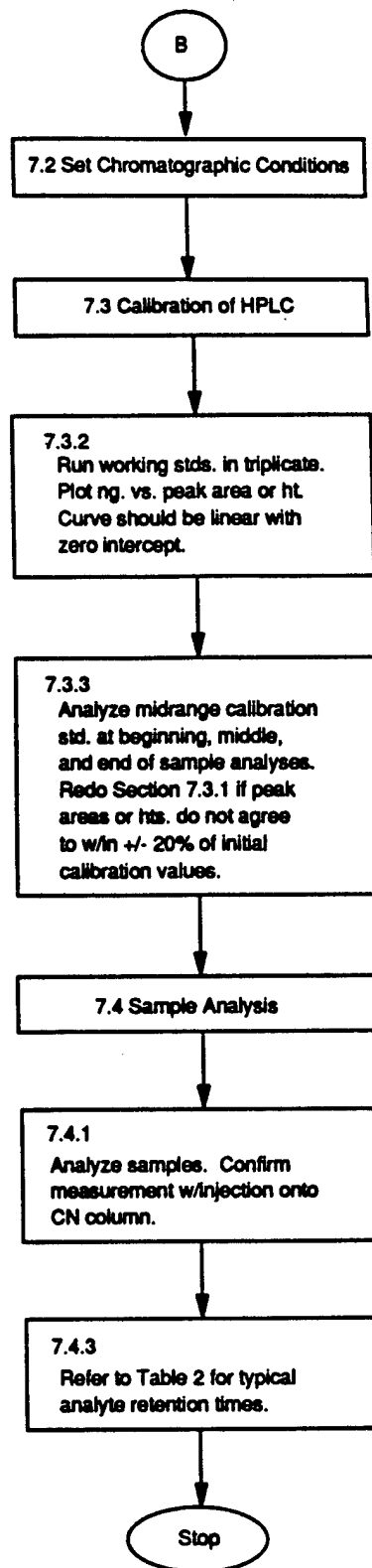
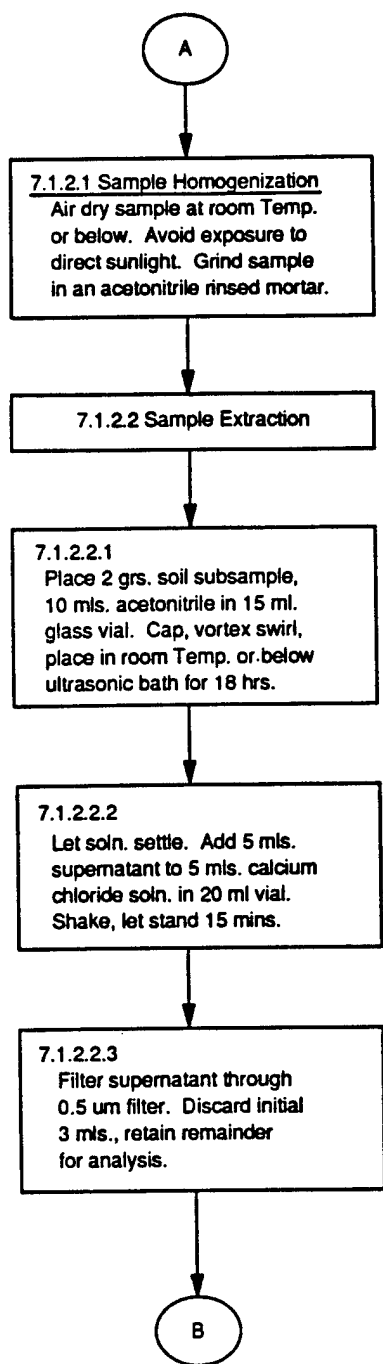


**FIGURE 1**  
**CHROMATOGRAMS FOR COLUMNS DESCRIBED IN SECTION 4.1.2.**  
**COURTESY OF U.S. ARMY CORPS OF ENGINEERS, OMAHA, NE.**

METHOD 8330  
NITROAROMATICS AND NITRAMINES BY HIGH  
PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)



METHOD 8330  
(continued)



**APPENDIX B5**

**AMMONIUM PICRATE ANALYSIS**

July 1990  
Revision: Final

**AMMONIUM PICRATE IN WATER AND WIPE SAMPLES BY  
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY**

# AMMONIUM PICRATE IN WATER AND WIPE SAMPLES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

## 1. Application

This method is suitable for the determination of ammonium picrate in water and wipe samples. Ammonium picrate is converted to picric acid and quantified in that form.

## 2. Summary of method

Ammonium picrate is extracted from water or wipe samples with solvent. The extract is concentrated and subjected to high performance liquid chromatography (HPLC) analysis using a reverse phase column and a dual channel ultraviolet detector.

## 3. Interferences

Any compounds that exhibit chemical and/or physical properties similar to the compounds of interest can interfere.

## 4. Apparatus

4.1 Concentrator apparatus, a Kuderna-Danish (K-D) concentrator, 500-mL capacity with a 3-ball Snyder column and a 10-mL graduated receiver tube and a 500-mL flask fitted with a 1-ball Snyder column.

4.2 Erlenmeyer flask, 500-mL with a ground glass stopper.

4.3 Evaporative concentrator, Organomation N-Evap, or equivalent.

4.4 Liquid chromatograph, Waters Associates ALC/GPC 204 liquid chromatograph equipped with a dual channel, variable wavelength detector, a model 6000A solvent-delivery system, a model 660 solvent flow programmer, model WISP 710A microprocessor and data module, or equivalents.

4.5 Liquid chromatographic column, a 3.9 mm i.d. by 300 mm long stainless steel column packed with 10  $\mu$ m particle size reverse phase material. Waters Associates  $\mu$ -Bonapak C<sub>18</sub> column packing or equivalent.

4.6 Separatory funnels, Squibb form, 1-L capacity, or equivalent.

4.7 Solvent clarification kit, Waters Associates 85113, or equivalent.  
1248E

6.1.3 Add 75 mL methylene chloride to the sample in the separatory funnel. Stopper and shake for 1 minute. Vent the pressure often. Allow the layers to separate and draw off the methylene chloride layer into a 250-mL Erlenmeyer flask.

6.1.4 Repeat the extraction of the water sample two more times, using 50-mL methylene chloride volumes each time. Combine all the organic extracts in the 250-mL Erlenmeyer, which contains the first extract.

6.1.5 Quantitatively transfer the extract to a 500-mL K-D apparatus fitted with a 3-ball Snyder column and a 20-mL receiver. Add a micro boiling chip and 4 mL of acetonitrile.

6.1.6 Place the apparatus on a hot-water bath (approximately 85°C). Reduce the volume of the extract to about 4 mL. Remove the K-D from the heat and allow it to cool.

6.1.7 Use the evaporative concentrator to reduce the volume of solvent to 0.8 to 0.9 mL by directing a stream of nitrogen onto the surface of the liquid while gently warming the receiver in a water bath.

6.1.8 Perform one method blank and three sample spikes at 40 ppb, 200 ppb, and 200 ppb with each batch of 20 samples.

6.1.9 Proceed to HPLC analysis (6.3).

## 6.2 Procedure for wipe samples

6.2.1 Add wipe sample to 40-mL vial.

6.2.2 Add 20 mL distilled water.

6.2.3 Shake 15 minutes using wrist-action shaker.

6.2.4 Decant water into 500-mL separatory funnel.

6.2.5 Repeat extraction twice, using 20 mL distilled water each time.

6.2.6 Add 440 mL distilled water to separator funnel.

6.2.7 Perform one method blank and three sample spikes of 20 ug, 100 ug, and 100 ug with each batch.

6.2.8 Proceed as in 6.1.2 under "Procedure for Water (Rinsate) Samples."

1248E

7.2 Water sample concentration is calculated as follows:

$$\frac{\text{ng/uL} \times \text{EV} \times 1 \text{ ug}}{1000 \text{ ng}} \div \text{SV} = \text{ug/L}$$

where:

ng/uL = picric acid concentration in extract, from LSF.  
EV = extract volume, in uL.  
SV = sample volume, in Liters.

Note that picric acid concentration must be converted to ammonium picrate concentration by using the ratios of molecular weights.

7.3 Wipe sample concentration (total ug) is calculated as follows:

$$\frac{\text{ng/uL} \times \text{EV} \times 1 \text{ ug}}{1000 \text{ ng}} = \text{total ug}$$

where:

ng/uL = picric acid concentration in extract, from LSF.  
EV = extract volume.

Note that picric acid concentration must be converted to ammonium picrate concentration by using the ratios of molecular weights.



**APPENDIX B6**

**EPA MODIFIED METHOD 5 SAMPLING TRAIN**

**EPA MODIFIED METHOD 5**  
**MODIFIED METHOD 5 SAMPLING TRAIN**

## METHOD 0010

### MODIFIED METHOD 5 SAMPLING TRAIN

#### 1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Destruction and Removal Efficiency (DRE) of semivolatile Principal Organic Hazardous Compounds (POHCs) from incineration systems (PHS, 1967). This method also may be used to determine particulate emission rates from stationary sources as per EPA Method 5 (see References at end of this method).

#### 2.0 SUMMARY OF METHOD

2.1 Gaseous and particulate pollutants are withdrawn from an emission source at an isokinetic sampling rate and are collected in a multicomponent sampling train. Principal components of the train include a high-efficiency glass- or quartz-fiber filter and a packed bed of porous polymeric adsorbent resin. The filter is used to collect organic-laden particulate materials and the porous polymeric resin to adsorb semivolatile organic species. Semivolatile species are defined as compounds with boiling points  $>100^{\circ}\text{C}$ .

2.2 Comprehensive chemical analyses of the collected sample are conducted to determine the concentration and identity of the organic materials.

#### 3.0 INTERFERENCES

3.1 Oxides of nitrogen ( $\text{NO}_x$ ) are possible interferents in the determination of certain water-soluble compounds such as dioxane, phenol, and urethane; reaction of these compounds with  $\text{NO}_x$  in the presence of moisture will reduce their concentration. Other possibilities that could result in positive or negative bias are (1) stability of the compounds in methylene chloride, (2) the formation of water-soluble organic salts on the resin in the presence of moisture, and (3) the solvent extraction efficiency of water-soluble compounds from aqueous media. Use of two or more ions per compound for qualitative and quantitative analysis can overcome interference at one mass. These concerns should be addressed on a compound-by-compound basis before using this method.

#### 4.0 APPARATUS AND MATERIALS

##### 4.1 Sampling train:

4.1.1 A schematic of the sampling train used in this method is shown in Figure 1. This sampling train configuration is adapted from EPA Method 5 procedures, and, as such, the majority of the required equipment

is identical to that used in EPA Method 5 determinations. The new components required are a condenser coil, and a sorbent module, which are used to collect semivolatile organic materials that pass through the glass- or quartz-fiber filter in the gas phase.

4.1.2 Construction details for the basic train components are given in APTD-0581 (see Martin, 1971, in Section 13.0, References); commercial models of this equipment are also available. Specifications for the sorbent module are provided in the following subsections. Additionally, the following subsections list changes to APTD-0581 and identify allowable train configuration modifications.

4.1.3 Basic operating and maintenance procedures for the sampling train are described in APTD-0576 (see Rom, 1972, in Section 13.0, References). As correct usage is important in obtaining valid results, all users should refer to APTD-0576 and adopt the operating and maintenance procedures outlined therein unless otherwise specified. The sampling train consists of the components detailed below.

4.1.3.1 Probe nozzle: Stainless steel (316) or glass with sharp, tapered (30° angle) leading edge. The taper shall be on the outside to preserve a constant I.D. The nozzle shall be buttonhook or elbow design and constructed from seamless tubing (if made of stainless steel). Other construction materials may be considered for particular applications. A range of nozzle sizes suitable for isokinetic sampling should be available in increments of 0.16 cm (1/16 in.), e.g., 0.32-1.27 cm (1/8-1/2 in.), or larger if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in Paragraph 9.1.

4.1.3.2 Probe liner: Borosilicate or quartz-glass tubing with a heating system capable of maintaining a gas temperature of  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ) at the exit end during sampling. (The tester may opt to operate the equipment at a temperature lower than that specified.) Because the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) are considered acceptable. Either borosilicate or quartz-glass probe liners may be used for stack temperatures up to about  $480^{\circ}\text{C}$  ( $900^{\circ}\text{F}$ ). Quartz liners shall be used for temperatures between 480 and  $900^{\circ}\text{C}$  (900 and  $1650^{\circ}\text{F}$ ). (The softening temperature for borosilicate is  $820^{\circ}\text{C}$  ( $1508^{\circ}\text{F}$ ), and for quartz  $1500^{\circ}\text{C}$  ( $2732^{\circ}\text{F}$ ).) Water-cooling of the stainless steel sheath will be necessary at temperatures approaching and exceeding  $500^{\circ}\text{C}$ .

4.1.3.3 Pitot tube: Type S, as described in Section 2.1 of EPA Method 2, or other appropriate devices (Vollaro, 1976). The pitot tube shall be attached to the probe to allow constant monitoring of the stack-gas velocity. The impact (high-pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see EPA Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of EPA Method 2.

complete organic module are not currently available, but may be assembled from commercially available laboratory glassware and a custom-fabricated sorbent trap. Details of two acceptable designs are shown in Figures 2 and 3 (the thermocouple well is shown in Figure 2).

4.1.3.8 Impinger train: To determine the stack-gas moisture content, four 500-mL impingers, connected in series with leak-free ground-glass joints, follow the knockout trap. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (1/2-in.) I.D. glass tube extending about 1.3 cm (1/2 in.) from the bottom of the outer cylinder. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of water or appropriate trapping solution. The third shall be empty or charged with a caustic solution, should the stack gas contain hydrochloric acid (HCl). The fourth shall contain a known weight of silica gel or equivalent desiccant.

4.1.3.9 Metering system: The necessary components are a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry-gas meter capable of measuring volume to within 1%, and related equipment, as shown in Figure 1. At a minimum, the pump should be capable of 4 cfm free flow, and the dry-gas meter should have a recording capacity of 0-999.9 cu ft with a resolution of 0.005 cu ft. Other metering systems capable of maintaining sampling rates within 10% of isokineticity and of determining sample volumes to within 2% may be used. The metering system must be used in conjunction with a pitot tube to enable checks of isokinetic sampling rates. Sampling trains using metering systems designed for flow rates higher than those described in APTD-0581 and APTD-0576 may be used, provided that the specifications of this method are met.

4.1.3.10 Barometer: Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby National Weather Service station, in which case the station value (which is the absolute barometric pressure) is requested and an adjustment for elevation differences between the weather station and sampling point is applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30-m (100 ft) elevation increase (vice versa for elevation decrease).

4.1.3.11 Gas density determination equipment: Temperature sensor and pressure gauge (as described in Sections 2.3 and 2.4 of EPA Method 2), and gas analyzer, if necessary (as described in EPA Method 3). The temperature sensor ideally should be permanently attached to the pitot tube or sampling probe in a fixed configuration such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal.

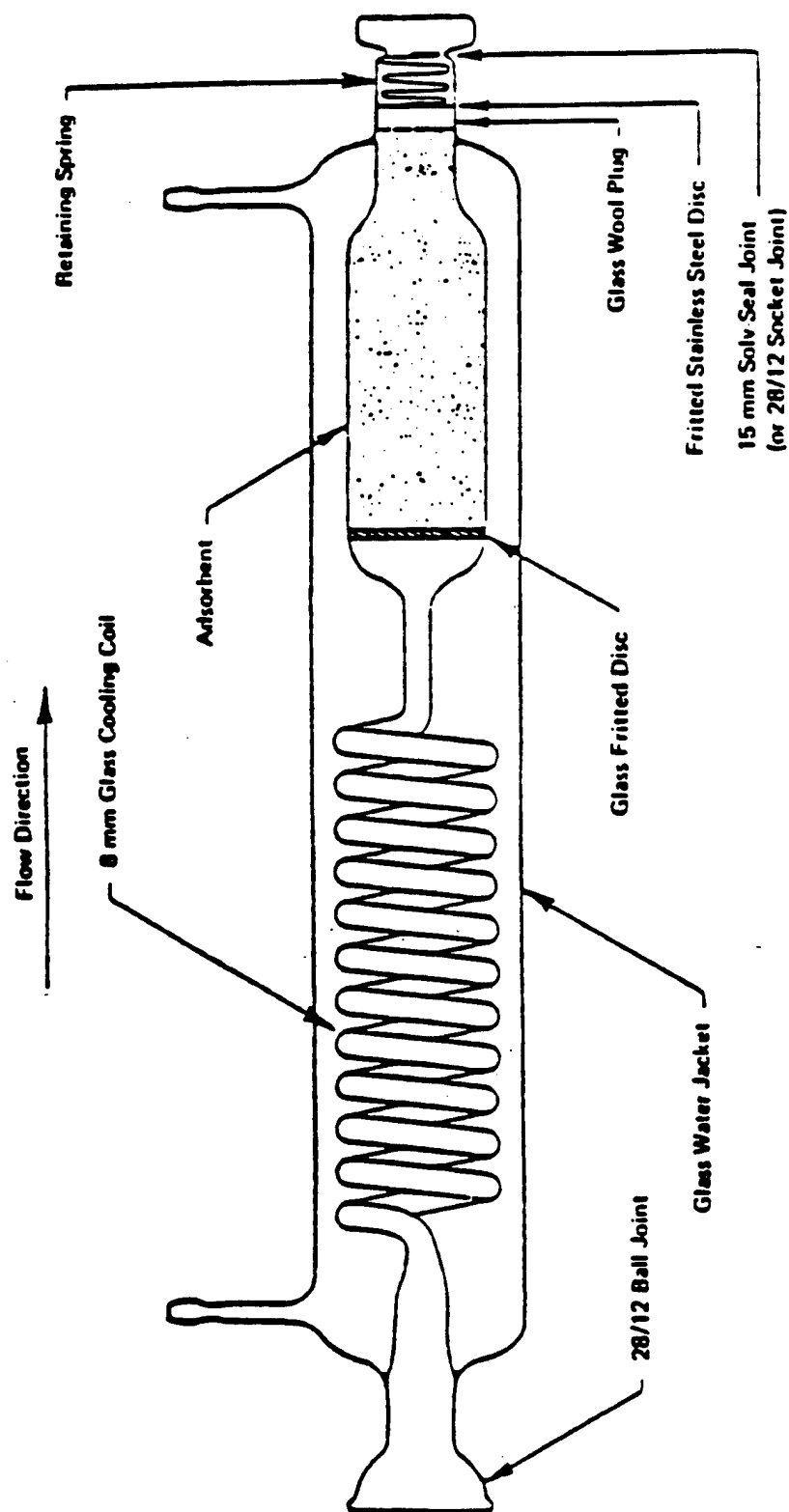


Figure 3. Adsorbent Sampling System.

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#### 4.2.8 Funnels: Glass, to aid in sample recovery.

4.3 Filters: Glass- or quartz-fiber filters, without organic binder, exhibiting at least 99.95% efficiency (<0.05% penetration) on 0.3- $\mu$ m dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D2986-71. Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO<sub>2</sub> or SO<sub>3</sub>, the filter material must be of a type that is unreactive to SO<sub>2</sub> or SO<sub>3</sub>. Reeve Angel 934 AH or Schleicher and Schuell #3 filters work well under these conditions.

4.4 Crushed ice: Quantities ranging from 10-50 lb may be necessary during a sampling run, depending on ambient air temperature.

4.5 Stopcock grease: Solvent-insoluble, heat-stable silicone grease. Use of silicone grease upstream of the module is not permitted, and amounts used on components located downstream of the organic module shall be minimized. Silicone grease usage is not necessary if screw-on connectors and Teflon sleeves or ground-glass joints are used.

4.6 Glass wool: Used to plug the unfritted end of the sorbent module. The glass-wool fiber should be solvent-extracted with methylene chloride in a Soxhlet extractor for 12 hr and air-dried prior to use.

### 5.0 REAGENTS

5.1 Adsorbent resin: Porous polymeric resin (XAD-2 or equivalent) is recommended. These resins shall be cleaned prior to their use for sample collection. Appendix A of this method should be consulted to determine appropriate precleaning procedure. For best results, resin used should not exhibit a blank of higher than 4 mg/kg of total chromatographable organics (TCO) (see Appendix B) prior to use. Once cleaned, resin should be stored in an airtight, wide-mouth amber glass container with a Teflon-lined cap or placed in one of the glass sorbent modules tightly sealed with Teflon film and elastic bands. The resin should be used within 4 wk of the preparation.

5.2 Silica gel: Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hr before using. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

5.3 Impinger solutions: Distilled organic-free water (Type II) shall be used, unless sampling is intended to quantify a particular inorganic gaseous species. If sampling is intended to quantify the concentration of additional species, the impinger solution of choice shall be subject to Administrator approval. This water should be prescreened for any compounds of interest. One hundred mL will be added to the specified impinger; the third impinger in the train may be charged with a basic solution (1 N sodium hydroxide or sodium acetate) to protect the sampling pump from acidic gases. Sodium acetate should be used when large sample volumes are anticipated because sodium hydroxide will react with carbon dioxide in aqueous media to form sodium carbonate, which may possibly plug the impinger.

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### **6.3 Preliminary field determinations:**

6.3.1 Select the sampling site and the minimum number of sampling points according to EPA Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and range of velocity heads using EPA Method 2. It is recommended that a leak-check of the pitot lines (see EPA Method 2, Section 3.1) be performed. Determine the stack-gas moisture content using EPA Approximation Method 4 or its alternatives to establish estimates of isokinetic sampling-rate settings. Determine the stack-gas dry molecular weight, as described in EPA Method 2, Section 3.6. If integrated EPA Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the sample run.

6.3.2 Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of EPA Method 2).

6.3.3 Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, to reduce the length of the probe, consider sampling from opposite sides of the stack.

6.3.4 A minimum of 3 dscm (105.9 dscf) of sample volume is required for the determination of the Destruction and Removal Efficiency (DRE) of POHCs from incineration systems. Additional sample volume shall be collected as necessitated by analytical detection limit constraints. To determine the minimum sample volume required, refer to sample calculations in Section 10.0.

6.3.5 Determine the total length of sampling time needed to obtain the identified minimum volume by comparing the anticipated average sampling rate with the volume requirement. Allocate the same time to all traverse points defined by EPA Method 1. To avoid timekeeping errors, the length of time sampled at each traverse point should be an integer or an integer plus one-half min.

6.3.6 In some circumstances (e.g., batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas-sample volumes. In these cases, the Administrator's approval must first be obtained.

### **6.4 Preparation of collection train:**

6.4.1 During preparation and assembly of the sampling train, keep all openings where contamination can occur covered with Teflon film or aluminum foil until just prior to assembly or until sampling is about to begin.



6.4.8 Turn on the sorbent module and condenser coil coolant recirculating pump and begin monitoring the sorbent module gas entry temperature. Ensure proper sorbent module gas entry temperature before proceeding and again before any sampling is initiated. It is extremely important that the XAD-2 resin temperature never exceed 50°C (122°F), because thermal decomposition will occur. During testing, the XAD-2 temperature must not exceed 20°C (68°F) for efficient capture of the semivolatile species of interest.

6.4.9 Turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize.

## 6.5 Leak-check procedures

### 6.5.1 Pre-test leak-check:

6.5.1.1 Because the number of additional intercomponent connections in the Semi-VOST train (over the M5 Train) increases the possibility of leakage, a pre-test leak-check is required.

6.5.1.2 After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 381-mm Hg (15-in. Hg) vacuum.

(NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.)

6.5.1.3 If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first attaching a carbon-filled leak-check impinger (shown in Figure 4) to the inlet of the filter holder (cyclone, if applicable) and then plugging the inlet and pulling a 381-mm Hg (15-in. Hg) vacuum. (Again, a lower vacuum may be used, provided that it is not exceeded during the test.) Then, connect the probe to the train and leak-check at about 25-mm Hg (1-in. Hg) vacuum; alternatively, leak-check the probe with the rest of the sampling train in one step at 381-mm Hg (15-in. Hg) vacuum. Leakage rates in excess of 4% of the average sampling rate or  $>0.00057 \text{ m}^3/\text{min}$  (0.02 cfm), whichever is less, are unacceptable.

6.5.1.4 The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with fine-adjust valve fully open and coarse-adjust valve completely closed. Partially open the coarse-adjust valve and slowly close the fine-adjust valve until the desired vacuum is reached. Do not reverse direction of the fine-adjust valve; this will cause water to back up into the organic module. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check, as shown below, and start over.

6.5.1.5 When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable). When the vacuum drops to 127 mm (5 in.) Hg or less, immediately close the coarse-adjust valve. Switch off the pumping system and reopen the fine-adjust valve. Do not reopen the fine-adjust valve until the coarse-adjust valve has been closed. This prevents the water in the impingers from being forced backward into the organic module and silica gel from being entrained backward into the third impinger.

#### 6.5.2 Leak-checks during sampling run:

6.5.2.1 If, during the sampling run, a component (e.g., filter assembly, impinger, or sorbent trap) change becomes necessary, a leak-check shall be conducted immediately after the interruption of sampling and before the change is made. The leak-check shall be done according to the procedure outlined in Paragraph 6.5.1, except that it shall be done at a vacuum greater than or equal to the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, the tester shall void the sampling run. (It should be noted that any "correction" of the sample volume by calculation reduces the integrity of the pollutant concentrations data generated and must be avoided.)

6.5.2.2 Immediately after a component change, and before sampling is reinitiated, a leak-check similar to a pre-test leak-check must also be conducted.

#### 6.5.3 Post-test leak-check:

6.5.3.1 A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done with the same procedures as those with the pre-test leak-check, except that it shall be conducted at a vacuum greater than or equal to the maximum value reached during the sampling run. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4% of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate, correct the sample volume (as shown in the calculation section of this method), and consider the data obtained of questionable reliability, or void the sampling run.

#### 6.6 Sampling-train operation:

6.6.1 During the sampling run, maintain an isokinetic sampling rate to within 10% of true isokinetic, unless otherwise specified by the Administrator. Maintain a temperature around the filter of  $120 \pm 14^\circ\text{C}$  ( $248 \pm 25^\circ\text{F}$ ) and a gas temperature entering the sorbent trap at a maximum of  $20^\circ\text{C}$  ( $68^\circ\text{F}$ ).

the 1990s, the number of people in the United States who are 65 years of age or older has increased by 50 percent, and the number of people 75 years of age or older has increased by 100 percent. The number of people 85 years of age or older has increased by 200 percent. The number of people 95 years of age or older has increased by 400 percent. The number of people 100 years of age or older has increased by 1,000 percent. The number of people 105 years of age or older has increased by 2,000 percent. The number of people 110 years of age or older has increased by 4,000 percent. The number of people 115 years of age or older has increased by 8,000 percent. The number of people 120 years of age or older has increased by 16,000 percent. The number of people 125 years of age or older has increased by 32,000 percent. The number of people 130 years of age or older has increased by 64,000 percent. The number of people 135 years of age or older has increased by 128,000 percent. The number of people 140 years of age or older has increased by 256,000 percent. The number of people 145 years of age or older has increased by 512,000 percent. The number of people 150 years of age or older has increased by 1,024,000 percent. The number of people 155 years of age or older has increased by 2,048,000 percent. The number of people 160 years of age or older has increased by 4,096,000 percent. The number of people 165 years of age or older has increased by 8,192,000 percent. The number of people 170 years of age or older has increased by 16,384,000 percent. The number of people 175 years of age or older has increased by 32,768,000 percent. The number of people 180 years of age or older has increased by 65,536,000 percent. The number of people 185 years of age or older has increased by 131,072,000 percent. The number of people 190 years of age or older has increased by 262,144,000 percent. The number of people 195 years of age or older has increased by 524,288,000 percent. The number of people 200 years of age or older has increased by 1,048,576,000 percent. The number of people 205 years of age or older has increased by 2,097,152,000 percent. The number of people 210 years of age or older has increased by 4,194,304,000 percent. The number of people 215 years of age or older has increased by 8,388,608,000 percent. The number of people 220 years of age or older has increased by 16,777,216,000 percent. The number of people 225 years of age or older has increased by 33,554,432,000 percent. The number of people 230 years of age or older has increased by 67,108,864,000 percent. The number of people 235 years of age or older has increased by 134,217,728,000 percent. The number of people 240 years of age or older has increased by 268,435,456,000 percent. The number of people 245 years of age or older has increased by 536,870,912,000 percent. The number of people 250 years of age or older has increased by 1,073,741,824,000 percent. The number of people 255 years of age or older has increased by 2,147,483,648,000 percent. The number of people 260 years of age or older has increased by 4,294,967,296,000 percent. The number of people 265 years of age or older has increased by 8,589,934,592,000 percent. The number of people 270 years of age or older has increased by 17,179,869,184,000 percent. The number of people 275 years of age or older has increased by 34,359,738,368,000 percent. The number of people 280 years of age or older has increased by 68,719,476,736,000 percent. The number of people 285 years of age or older has increased by 137,438,953,472,000 percent. The number of people 290 years of age or older has increased by 274,877,906,944,000 percent. The number of people 295 years of age or older has increased by 549,755,813,888,000 percent. The number of people 300 years of age or older has increased by 1,099,511,627,776,000 percent. The number of people 305 years of age or older has increased by 2,199,023,255,552,000 percent. The number of people 310 years of age or older has increased by 4,398,046,511,104,000 percent. The number of people 315 years of age or older has increased by 8,796,093,022,208,000 percent. The number of people 320 years of age or older has increased by 17,592,186,044,416,000 percent. The number of people 325 years of age or older has increased by 35,184,372,088,832,000 percent. The number of people 330 years of age or older has increased by 70,368,744,177,664,000 percent. The number of people 335 years of age or older has increased by 140,737,488,355,328,000 percent. The number of people 340 years of age or older has increased by 281,474,976,710,656,000 percent. The number of people 345 years of age or older has increased by 562,949,953,421,312,000 percent. The number of people 350 years of age or older has increased by 1,125,899,906,842,624,000 percent. The number of people 355 years of age or older has increased by 2,251,799,813,685,248,000 percent. The number of people 360 years of age or older has increased by 4,503,599,627,370,496,000 percent. The number of people 365 years of age or older has increased by 9,007,199,254,740,992,000 percent. The number of people 370 years of age or older has increased by 18,014,398,509,481,984,000 percent. The number of people 375 years of age or older has increased by 36,028,797,018,963,968,000 percent. The number of people 380 years of age or older has increased by 72,057,594,037,927,936,000 percent. The number of people 385 years of age or older has increased by 144,115,188,075,855,872,000 percent. The number of people 390 years of age or older has increased by 288,230,376,151,711,744,000 percent. The number of people 395 years of age or older has increased by 576,460,752,303,423,488,000 percent. The number of people 400 years of age or older has increased by 1,152,921,504,606,846,976,000 percent. The number of people 405 years of age or older has increased by 2,305,843,009,213,693,952,000 percent. The number of people 410 years of age or older has increased by 4,611,686,018,427,387,904,000 percent. The number of people 415 years of age or older has increased by 9,223,372,036,854,775,808,000 percent. The number of people 420 years of age or older has increased by 18,446,744,073,709,551,616,000 percent. The number of people 425 years of age or older has increased by 36,893,488,147,419,103,232,000 percent. The number of people 430 years of age or older has increased by 73,786,976,294,838,206,464,000 percent. The number of people 435 years of age or older has increased by 147,573,952,589,676,412,928,000 percent. The number of people 440 years of age or older has increased by 295,147,905,179,352,825,856,000 percent. The number of people 445 years of age or older has increased by 590,295,810,358,705,651,712,000 percent. The number of people 450 years of age or older has increased by 1,180,591,620,717,411,303,424,000 percent. The number of people 455 years of age or older has increased by 2,361,183,241,434,822,606,848,000 percent. The number of people 460 years of age or older has increased by 4,722,366,482,869,645,213,696,000 percent. The number of people 465 years of age or older has increased by 9,444,732,965,739,290,427,392,000 percent. The number of people 470 years of age or older has increased by 18,889,465,931,478,580,854,784,000 percent. The number of people 475 years of age or older has increased by 37,778,931,862,957,161,709,568,000 percent. The number of people 480 years of age or older has increased by 75,557,863,725,914,323,419,136,000 percent. The number of people 485 years of age or older has increased by 151,115,727,451,828,646,838,272,000 percent. The number of people 490 years of age or older has increased by 302,231,454,903,657,293,676,544,000 percent. The number of people 495 years of age or older has increased by 604,462,909,807,314,587,353,088,000 percent. The number of people 500 years of age or older has increased by 1,208,925,819,614,629,174,706,176,000 percent. The number of people 505 years of age or older has increased by 2,417,851,639,229,258,349,412,352,000 percent. The number of people 510 years of age or older has increased by 4,835,703,278,458,516,698,824,704,000 percent. The number of people 515 years of age or older has increased by 9,671,406,556,917,033,397,649,408,000 percent. The number of people 520 years of age or older has increased by 19,342,813,113,834,066,795,298,816,000 percent. The number of people 525 years of age or older has increased by 38,685,626,227,668,133,590,597,632,000 percent. The number of people 530 years of age or older has increased by 77,371,252,455,336,267,181,195,264,000 percent. The number of people 535 years of age or older has increased by 154,742,504,910,672,534,362,390,528,000 percent. The number of people 540 years of age or older has increased by 309,485,009,821,345,068,724,781,056,000 percent. The number of people 545 years of age or older has increased by 618,970,019,642,690,137,449,562,112,000 percent. The number of people 550 years of age or older has increased by 1,237,940,039,285,380,274,899,124,224,000 percent. The number of people 555 years of age or older has increased by 2,475,880,078,570,760,549,798,248,448,000 percent. The number of people 560 years of age or older has increased by 4,951,760,157,141,521,099,596,496,896,000 percent. The number of people 565 years of age or older has increased by 9,903,520,314,283,042,199,193,993,792,000 percent. The number of people 570 years of age or older has increased by 19,807,040,628,566,084,398,387,

### Schematic of Stack Cross Section

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Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the organic module and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the organic module.

7.1.3 Cap the filter-holder outlet and the inlet to the organic module. Separate the sorbent trap section of the organic module from the condensate knockout trap and the gas-conditioning section. Cap all organic module openings. Disconnect the organic-module knockout trap from the impinger train inlet and cap both of these openings. Ground-glass stoppers, Teflon caps, or caps of other inert materials may be used to seal all openings.

7.1.4 Transfer the probe, the filter, the organic-module components, and the impinger/condenser assembly to the cleanup area. This area should be clean and protected from the weather to minimize sample contamination or loss.

7.1.5 Save a portion of all washing solutions (methanol/methylene chloride, Type II water) used for cleanup as a blank. Transfer 200 mL of each solution directly from the wash bottle being used and place each in a separate, prelabeled glass sample container.

7.1.6 Inspect the train prior to and during disassembly and note any abnormal conditions.

## 7.2 Sample containers:

7.2.1 Container no. 1: Carefully remove the filter from the filter holder and place it in its identified Petri dish container. Use a pair or pairs of tweezers to handle the filter. If it is necessary to fold the filter, ensure that the particulate cake is inside the fold. Carefully transfer to the Petri dish any particulate matter or filter fibers that adhere to the filter-holder gasket, using a dry nylon bristle brush or sharp-edged blade, or both. Label the container and seal with 1-in.-wide Teflon tape around the circumference of the lid.

7.2.2 Container no. 2: Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components first with methanol/methylene chloride (1:1 v/v) into a glass container. Distilled water may also be used. Retain a water and solvent blank and analyze in the same manner as with the samples. Perform rinses as follows:

7.2.2.1 Carefully remove the probe nozzle and clean the inside surface by rinsing with the solvent mixture (1:1 v/v methanol/methylene chloride) from a wash bottle and brushing with a nylon bristle brush. Brush until the rinse shows no visible particles; then make a final rinse of the inside surface with the solvent mix. Brush and rinse the inside parts of the Swagelok fitting with the solvent mix in a similar way until no visible particles remain.

7.2.4 Container no. 4: Measure the volume of condensate collected in the condensate knockout section of the organic module to within +1 mL by using a graduated cylinder or by weighing to within +0.5 g using a triple-beam balance. Record the volume or weight of liquid present and note any discoloration or film in the liquid catch. Transfer this liquid to a prelabeled glass sample container. Inspect the back half of the filter housing and the gas-conditioning section of the organic module. If condensate is observed, transfer it to a graduated or weighing bottle and measure the volume, as described above. Add this material to the condensate knockout-trap catch.

7.2.5 Container no. 5: All sampling train components located between the high-efficiency glass- or quartz-fiber filter and the first wet impinger or the final condenser system (including the heated Teflon line connecting the filter outlet to the condenser) should be thoroughly rinsed with methanol/methylene chloride (1:1 v/v) and the rinsings combined. This rinse shall be separated from the condensate. If the spent resin is transferred from the sorbent trap to a separate sample container for transport, the sorbent trap shall be thoroughly rinsed until all sample-wetted surfaces appear clean. Visible films should be removed by brushing. Whenever train components are brushed, the brush should be subsequently rinsed with solvent mixture and the rinsings added to this container.

7.2.6 Container no. 6: Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere strongly to the impinger wall. Because the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the container and its contents to 0.5 g or better.

### 7.3 Impinger water:

7.3.1 Make a notation of any color or film in the liquid catch. Measure the liquid in the first three impingers to within +1 mL by using a graduated cylinder or by weighing it to within +0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

7.3.2 Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Paragraph 4.1.3.7). Amber glass containers should be used for storage of impinger catch, if required.

7.3.3 If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

8.1.3 Impinger: Spike the sample with the surrogate standards; measure and record the volume and transfer to a separatory funnel. Proceed as described in Paragraph 8.1.2.

8.1.4 XAD-2: Spike the resin directly with the surrogate standards. Transfer the resin to the all-glass thimbles by the following procedure (care should be taken so as not to contaminate the thimble by touching it with anything other than tweezers or other solvent-rinsed mechanical holding devices). Suspend the XAD-2 module directly over the thimble. The glass frit of the module (see Figure 2) should be in the up position. The thimble is contained in a clean beaker, which will serve to catch the solvent rinses. Using a Teflon squeeze bottle, flush the XAD-2 into the thimble. Thoroughly rinse the glass module with solvent into the beaker containing the thimble. Add the XAD-2 glass-wool plug to the thimble. Cover the XAD-2 in the thimble with a precleaned glass-wool plug sufficient to prevent the resin from floating into the solvent reservoir of the extractor. If the resin is wet, effective extraction can be accomplished by loosely packing the resin in the thimble. If a question arises concerning the completeness of the extraction, a second extraction, without a spike, is advised. The thimble is placed in the extractor and the rinse solvent contained in the beaker is added to the solvent reservoir. Additional solvent is added to make the reservoir approximately two-thirds full. Add Teflon boiling chips and assemble the apparatus. Adjust the heat source to cause the extractor to cycle 5-6 times per hr. Extract the resin for 16 hr. Transfer the solvent and three 10-mL rinses of the reservoir to a K-D and concentrate as described in Paragraph 8.1.2.

8.1.5 Particulate filter (and cyclone catch): If particulate loading is to be determined, weigh the filter (and cyclone catch, if applicable). The particulate filter (and cyclone catch, if applicable) is transferred to the glass thimble and extracted simultaneously with the XAD-2 resin.

8.1.6 Train solvent rinses: All train rinses (i.e., probe, impinger, filter housing) using the extraction solvent and methanol are returned to the laboratory as a single sample. If the rinses are contained in more than one container, the intended spike is divided equally among the containers proportioned from a single syringe volume. Transfer the rinse to a separatory funnel and add a sufficient amount of organic-free water so that the methylene chloride becomes immiscible and its volume no longer increases with the addition of more water. The extraction and concentration steps are then performed as described in Paragraph 8.1.2.

## 8.2 Sample analysis:

8.2.1 The primary analytical tool for the measurement of emissions from hazardous waste incinerators is GC/MS using fused-silica capillary GC columns, as described in Method 8270 in Chapter Four of this manual. Because of the nature of GC/MS instrumentation and the cost associated

### 9.3 Metering system:

9.3.1 Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0576. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to correct the gas meter dial readings mathematically to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: Make a 10-min calibration run at  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm); at the end of the run, take the difference of the measured wet-test and dry-gas meter volumes and divide the difference by 10 to get the leak rate. The leak rate should not exceed  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm).

9.3.2 After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single intermediate orifice setting (based on the previous field test). The vacuum shall be set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576.

9.3.3 Leak-check of metering system: That portion of the sampling train from the pump to the orifice meter (see Figure 1) should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 6): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13-18 cm (5-7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 min. A loss of pressure on the manometer indicates a leak in the meter box. Leaks, if present, must be corrected.

NOTE: If the dry-gas-meter coefficient values obtained before and after a test series differ by >5%, either the test series shall be voided or calculations for test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

9.4 Probe heater: The probe-heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

9.5 Temperature gauges: Each thermocouple must be permanently and uniquely marked on the casting; all mercury-in-glass reference thermometers must conform to ASTM E-1 63C or 63F specifications. Thermocouples should be calibrated in the laboratory with and without the use of extension leads. If extension leads are used in the field, the thermocouple readings at ambient air temperatures, with and without the extension lead, must be noted and recorded. Correction is necessary if the use of an extension lead produces a change  $>1.5\%$ .

9.5.1 Impinger, organic module, and dry-gas meter thermocouples: For the thermocouples used to measure the temperature of the gas leaving the impinger train and the XAD-2 resin bed, three-point calibration at ice-water, room-air, and boiling-water temperatures is necessary. Accept the thermocouples only if the readings at all three temperatures agree to  $\pm 2^{\circ}\text{C}$  ( $3.6^{\circ}\text{F}$ ) with those of the absolute value of the reference thermometer.

9.5.2 Probe and stack thermocouple: For the thermocouples used to indicate the probe and stack temperatures, a three-point calibration at ice-water, boiling-water, and hot-oil-bath temperatures must be performed; it is recommended that room-air temperature be added, and that the thermometer and the thermocouple agree to within  $1.5\%$  at each of the calibration points. A calibration curve (equation) may be constructed (calculated) and the data extrapolated to cover the entire temperature range suggested by the manufacturer.

9.6 Barometer: Adjust the barometer initially and before each test series to agree to within  $\pm 25$  mm Hg ( $0.1$  in. Hg) of the mercury barometer or the corrected barometric pressure value reported by a nearby National Weather Service Station (same altitude above sea level).

9.7 Triple-beam balance: Calibrate the triple-beam balance before each test series, using Class-S standard weights; the weights must be within  $\pm 0.5\%$  of the standards, or the balance must be adjusted to meet these limits.

## 10.0 CALCULATIONS

10.1 Carry out calculations. Round off figures after the final calculation to the correct number of significant figures.

### 10.2 Nomenclature:

$A_n$  = Cross-sectional area of nozzle,  $\text{m}^2$  ( $\text{ft}^2$ ).

$B_{ws}$  = Water vapor in the gas stream, proportion by volume.

$C_d$  = Type S pitot tube coefficient (nominally  $0.84 \pm 0.02$ ), dimensionless.

$I$  = Percent of isokinetic sampling.



$\rho_w$  = Density of water, 0.9982 g/mL (0.002201 lb/mL).

$\theta$  = Total sampling time, min.

$\theta_1$  = Sampling time interval from the beginning of a run until the first component change, min.

$\theta_i$  = Sampling time interval between two successive component changes, beginning with the interval between the first and second changes, min.

$\theta_p$  = Sampling time interval from the final ( $n^{\text{th}}$ ) component change until the end of the sampling run, min.

13.6 = Specific gravity of mercury.

60 = sec/min.

100 = Conversion to percent.

10.3 Average dry-gas-meter temperature and average orifice pressure drop: See data sheet (Figure 5, above).

10.4 Dry-gas volume: Correct the sample measured by the dry-gas meter to standard conditions (20°C, 760 mm Hg [68°F, 29.92 in. Hg]) by using Equation 1:

$$V_{m(\text{std})} = V_m \gamma \frac{T_{\text{std}}}{T_m} \frac{P_{\text{bar}} + \Delta H/13.6}{P_{\text{std}}} = K_1 V_m \gamma \frac{P_{\text{bar}} + \Delta H/13.6}{T_m} \quad (1)$$

where:

$K_1$  = 0.3858 K/mm Hg for metric units, or

$K_1$  = 17.64°R/in. Hg for English units.

It should be noted that Equation 1 can be used as written, unless the leakage rate observed during any of the mandatory leak-checks (i.e., the post-test leak-check or leak-checks conducted prior to component changes) exceeds  $L_a$ . If  $L_p$  or  $L_i$  exceeds  $L_a$ , Equation 1 must be modified as follows:

- a. Case I (no component changes made during sampling run): Replace  $V_m$  in Equation 1 with the expression:

$$V_m - (L_p - L_a)$$

## 10.8 Isokinetic variation:

### 10.8.1 Calculation from raw data:

$$I = \frac{100 T_s [K_3 F_{1c} + (V_m/T_m) (P_{bar} + \Delta H/13.6)]}{608 V_s P_s A_n} \quad (4)$$

where:

$K_3 = 0.003454 \text{ mm Hg-m}^3/\text{mL-K}$  for metric units, or  
 $K_3 = 0.002669 \text{ in. Hg-ft}^3/\text{mL-}^\circ\text{R}$  for English units.

### 10.8.2 Calculation for intermediate values:

$$I = \frac{T_s V_m(\text{std}) P_{\text{std}}^{100}}{T_{\text{std}} V_s A_n P_s^{60(1-B_{ws})}} \quad (5)$$

$$= K_4 \frac{T_s V_m(\text{std})}{P_s V_s A_n^{60(1-B_{ws})}}$$

where:

$K_4 = 4.320$  for metric units, or  
 $K_4 = 0.09450$  for English units.

10.8.3 Acceptable results: If  $90\% \leq I \leq 110\%$ , the results are acceptable. If the results are low in comparison with the standard and  $I$  is beyond the acceptable range, or if  $I$  is less than 90%, the Administrator may opt to accept the results.

10.9 To determine the minimum sample volume that shall be collected, the following sequence of calculations shall be used.

10.9.1 From prior analysis of the waste feed, the concentration of POHCs introduced into the combustion system can be calculated. The degree of destruction and removal efficiency that is required is used to determine the maximum amount of POHC allowed to be present in the effluent. This may be expressed as:

$$\frac{(WF) (POHC_1 \text{ conc}) (100-\%DRE)}{100} = \text{Max POHC}_1 \text{ Mass} \quad (6)$$

where:

WF = mass flow rate of waste feed per hr, g/hr (lb/hr).

POHC<sub>1</sub> = concentration of Principal Organic Hazardous Compound (wt %) introduced into the combustion process.

where:

CPOHC = concentration of POHC as analyzed by Method 8270.

2) Sum the amount of POHC found in all samples associated with a single train.

Total (ug) = XAD-2 (ug) + condensate (ug) + rinses (ug) + impinger (ug) (10)

3) Divide the total ug found by the volume of stack gas sampled (m<sup>3</sup>).

(Total ug)/(train sample volume) = concentration of POHC (ug/m<sup>3</sup>) (11)

## 11.0 QUALITY CONTROL

11.1 Sampling: See EPA Manual 600/4-77-027b for Method 5 quality control.

11.2 Analysis: The quality assurance program required for this study includes the analysis of field and method blanks, procedure validations, incorporation of stable labeled surrogate compounds, quantitation versus stable labeled internal standards, capillary column performance checks, and external performance tests. The surrogate spiking compounds selected for a particular analysis are used as primary indicators of the quality of the analytical data for a wide range of compounds and a variety of sample matrices. The assessment of combustion data, positive identification, and quantitation of the selected compounds are dependent on the integrity of the samples received and the precision and accuracy of the analytical methods employed. The quality assurance procedures for this method are designed to monitor the performance of the analytical method and to provide the required information to take corrective action if problems are observed in laboratory operations or in field sampling activities.

11.2.1 Field Blanks: Field blanks must be submitted with the samples collected at each sampling site. The field blanks include the sample bottles containing aliquots of sample recovery solvents, unused filters, and resin cartridges. At a minimum, one complete sampling train will be assembled in the field staging area, taken to the sampling area, and leak-checked at the beginning and end of the testing (or for the same total number of times as the actual test train). The filter housing and probe of the blank train will be heated during the sample test. The train will be recovered as if it were an actual test sample. No gaseous sample will be passed through the sampling train.

11.2.2 Method blanks: A method blank must be prepared for each set of analytical operations, to evaluate contamination and artifacts that can be derived from glassware, reagents, and sample handling in the laboratory.

11.2.3 Refer to Method 8270 for additional quality control considerations.

6. Shigehara, R.T., Adjustments in the EPA Nomography for Different Pitot Tube Coefficients and Dry Molecular Weights, Stack Sampling News, 2:4-11 (October 1974).

7. U.S. Environmental Protection Agency, CFR 40 Part 60, Appendix A, Methods 1-5.

8. Vollaro, R.F., A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities, Research Triangle Park, NC, U.S. Environmental Protection Agency, Emissions Measurement Branch, November 1976 (unpublished paper).

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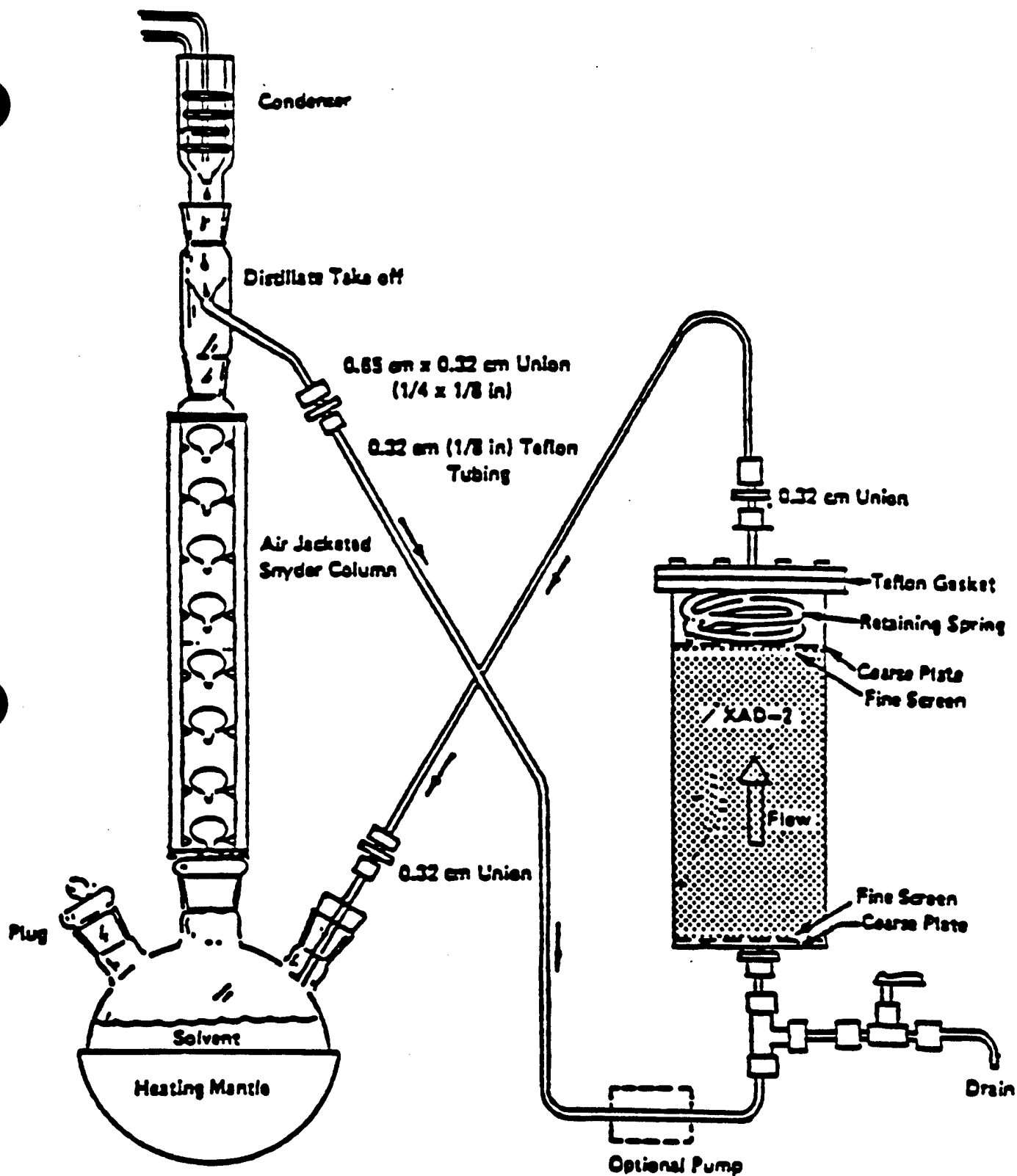


Figure A-1. XAD-2 cleanup extraction apparatus.

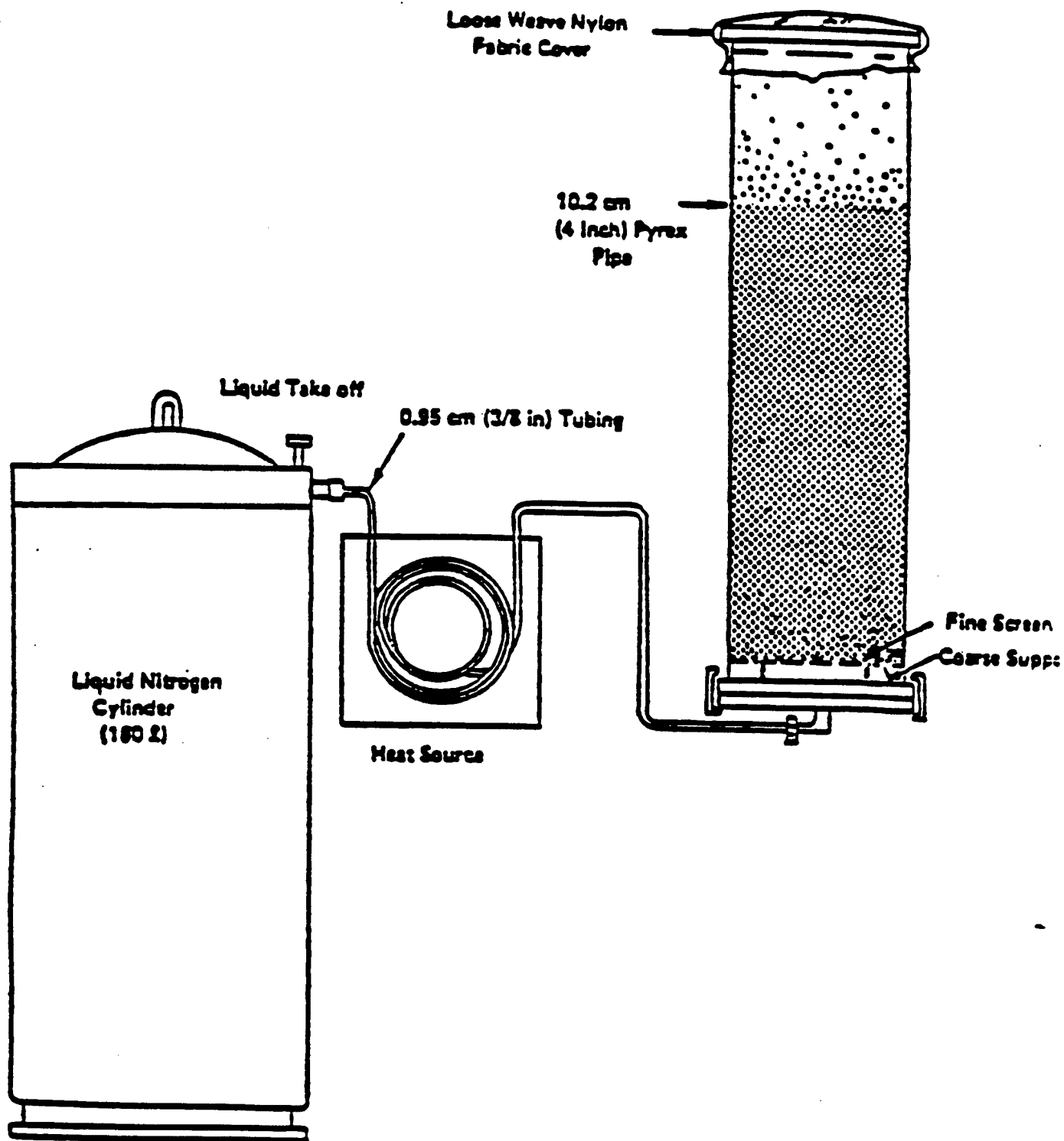


Figure A-2. XAD-2 fluidized-bed drying apparatus.

resin will float out unless well plugged.) The thimble containing the resin is extracted for 24 hr with 200-mL of pesticide-grade methylene chloride (Burdick and Jackson pesticide-grade or equivalent purity). The 200-mL extract is reduced in volume to 10-mL using a Kuderna-Danish concentrator and/or a nitrogen evaporation stream. Five uL of that solution are analyzed by gas chromatography using the TCO analysis procedure. The concentrated solution should not contain >20 ug/mL of TCO extracted from the XAD-2. This is equivalent to 10 ug/g of TCO in the XAD-2 and would correspond to 1.3 mg of TCO in the extract of the 130-g XAD-2 module. Care should be taken to correct the TCO data for a solvent blank prepared (200 mL reduced to 10 mL) in a similar manner.

4.4.2 Experimental: Use the TCO analysis conditions described in the revised Level 1 manual (EPA 600/7-78-201).

4.5 GC/MS Screen: The extract, as prepared in paragraph 4.4.1, is subjected to GC/MS analysis for each of the individual compounds of interest. The GC/MS procedure is described in Chapter Four, Method 8270. The extract is screened at the MDL of each compound. The presence of any compound at a concentration >25 ug/mL in the concentrated extract will require the XAD-2 to be recleaned by repeating the methylene chloride step.

4.6 Methodology for residual gravimetric determination: After the TCO value and GC/MS data are obtained for the resin batch by the above procedures, dry the remainder of the extract in a tared vessel. There must be <0.5 mg residue registered or the batch of resin will have to be extracted with fresh methylene chloride again until it meets this criterion. This level corresponds to 25 ug/g in the XAD-2, or about 3.25 mg in a resin charge of 130 g.

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will not elute from the column at all and thus will not be reported. Consequently, the organic content of the sample as reported is a lower bound and should be regarded as such.

1.6.2 Calibration limitations: Quantitation is based on calibration with n-decane. Data should therefore be reported as, e.g., mg C8/m<sup>3</sup> as n-decane. Since response varies linearly with carbon number (over a wide range the assumption may involve a 20% error), it is clear that heptane (C7) detected in a sample and quantitated as decane will be overestimated. Likewise, hexadecane (C16) quantitated as decane will be underestimated. From previous data, it is estimated the error involved is on the order of 6-7%.

1.6.3 Detection limitations: The sensitivity of the flame ionization detector varies from compound to compound. However, n-alkanes have a greater response than other classes. Consequently, using an n-alkane as a calibrant and assuming equal responses of all other compounds tends to give low reported values.

## 2.0 SUMMARY OF METHOD

2.1 A mL aliquot of all 10-mL concentrates is disbursed for GC-TCO analysis. With boiling point-retention time and response-amount calibration curves, the data (peak retention times and peak areas) are interpreted by first summing peak areas in the ranges obtained from the boiling point-retention time calibration. Then, with the response-amount calibration curve, the area sums are converted to amounts of material in the reported boiling point ranges.

2.2 After the instrument is set up, the boiling point-retention time calibration is effected by injecting a mixture of n-C7 through n-C16 hydrocarbons and operating the standard temperature program. Response-quantity calibrations are accomplished by injecting n-decane in n-pentane standards and performing the standard temperature program.

### 2.3 Definitions

2.3.1 GC: Gas chromatography or gas chromatograph.

2.3.2 C7-C16 n-alkanes: Heptane through hexadecane.

2.3.3 GCA temperature program: 4 min isothermal at 60°C, 10°C/min from 60° to 220°C.

2.3.4 TRW temperature program: 5 min isothermal at room temperature, then program from 30°C to 250°C at 15°C/min.

## 3.0 INTERFERENCES

Not applicable.

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5.2 Methylene chloride: "Distilled-in-Glass" (reg. trademark) or "Nanograde" (reg. trademark) for syringe cleaning.

## 6.0 SAMPLING HANDLING AND PRESERVATION

6.1 The extracts are concentrated in a Kuderna-Danish evaporator to a volume less than 10 mL. The concentrate is then quantitatively transferred to a 10-mL volumetric flask and diluted to volume. A 1-mL aliquot is taken for both this analysis and possible subsequent GC/MS analysis and set aside in the sample bank. For each GC-TCO analysis, obtain the sample sufficiently in advance to allow it to warm to room temperature. For example, after one analysis is started, return that sample to the sample bank and take the next sample.

## 7.0 PROCEDURES

7.1 Setup and checkout: Each day, the operator will verify the following:

7.1.1 That supplies of carrier gas, air and hydrogen are sufficient, i.e., that each tank contains  $> 100$  psig.

7.1.2 That, after replacement of any gas cylinder, all connections leading to the chromatograph have been leak-checked.

7.1.3 That the carrier gas flow rate is  $30 \pm 2$  mL/min, the hydrogen flow rate is  $30 \pm 2$  mL/min, and the air flow rate is  $300 \pm 20$  mL/min.

7.1.4 That the electrometer is functioning properly.

7.1.5 That the recorder and integrator are functioning properly.

7.1.6 That the septa have been leak-checked (leak-checking is effected by placing the soap bubble flow meter inlet tube over the injection port adaptors), and that no septum will be used for more than 20 injections.

7.1.7 That the list of samples to be run is ready.

7.2 Retention time calibration:

7.2.1 To obtain the temperature ranges for reporting the results of the analyses, the chromatograph is given a normal boiling point-retention time calibration. The n-alkanes, their boiling points, and data reporting ranges are given in the table below:

### 7.3 Response calibration:

7.3.1 For the purposes of a Level 1 analysis, response-quantity calibration with n-decane is adequate. A 10-uL volume of n-decane is injected into a tared 10 mL volumetric flask. The weight injected is obtained and the flask is diluted to the mark with n-pentane. This standard contains about 730 ng n-decane per uL n-pentane. The exact concentration depends on temperature, so that a weight is required. Two serial tenfold dilutions are made from this standard, giving standards at about 730, 73, and 7.3 ng n-decane per uL n-pentane, respectively.

7.3.2 Procedure for response calibration: This calibration is performed at the start of an analytical program and monthly thereafter. The most concentrated standard is injected once each day. Any change in calibration necessitates a full calibration with new standards. Standards are stored in the refrigerator locker and are made up monthly.

7.3.2.1 Verify that the instrument is set up properly.

7.3.2.2 Set electrometer at  $1 \times 10^{-10}$  A/mV.

7.3.2.3 Inject 1 uL of the highest concentration standard.

7.3.2.4 Run standard temperature program as specified above.

7.3.2.5 Clean syringe.

7.3.2.6 Make repeated injections of all three standards until the relative standard deviations of the areas of each standard are  $\leq 5\%$ .

### 7.4 Sample analysis procedure:

7.4.1 The following apparatus is required:

7.4.1.1 Gas chromatograph set up and working.

7.4.1.2 Recorder, integrator working.

7.4.1.3 Syringe and syringe cleaning apparatus.

7.4.1.4 Parameters: Electrometer setting is  $1 \times 10^{-10}$  A/mV; recorder is set at 0.5 in./min and 1 mV full-scale.

7.4.2 Steps in the procedure are:

7.4.2.1 Label chromatogram with the data, sample number, etc.

7.7.1.2 Plot average retention times as abscissae versus normal boiling points as ordinates.

7.7.1.3 Draw in calibration curve.

7.7.1.4 Locate and record retention times corresponding to boiling ranges 90-100, 110-140, 140-160, 160-180, 180-200, 200-220, 220-240, 240-260, 260-280, 280-300°C.

7.7.2 Response-amount calibration: The required data for this calibration are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.2.1 Average the area responses of each standard and calculate relative standard deviations.

7.7.2.2 Plot response (uV·sec) as ordinate versus ng/uL as abscissa.

7.7.2.3 Draw in the curve. Perform least squares regression and obtain slope (uV·sec·uL/ng).

7.7.3 Total C7-C16 hydrocarbons analysis: The required data for this calculation are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.3.1 Sum the areas of all peaks within the retention time range of interest.

7.7.3.2 Convert this area (uV·sec) to ng/uL by dividing by the weight response for n-decane (uV·sec·uL/ng).

7.7.3.3 Multiply this weight by the total concentrate volume (10 mL) to get the weight of the C7-C16 hydrocarbons in the sample.

7.7.3.4 Using the volume of gas sampled or the total weight of sample acquired, convert the result of Step 7.7.3.3 above to ug/m<sup>3</sup>.

7.7.3.5 If the value of total C7-C16 hydrocarbons from Step 7.7.3.4 above exceeds 75 ug/m<sup>3</sup>, calculate individual hydrocarbon concentrations in accordance with the instructions in Paragraph 7.7.5.5 below.

7.7.4 Individual C7-C16 n-Alkane Equivalent Analysis: The required data from the analyses are on the chromatogram and on the data sheet. The data reduction is performed as follows:

7.7.4.1 Sum the areas of peaks in the proper retention time ranges.

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**APPENDIX B7**

**EPA WIPE SAMPLING TECHNIQUE**

## **EPA WIPE SAMPLING TECHNIQUE**

## **SECTION 13**

### **SPECIALIZED SAMPLING TECHNIQUES**

#### **13.0 GENERAL**

This section discusses several specialized sampling techniques that have been used by contractors on hazardous waste sites. The reader may develop other techniques for specific site needs. In those cases and in cases where the techniques listed here are modified for use on a specific site, careful documentation of the exact procedures used should be provided. This section does not discuss analytical techniques, since analytical methods would vary depending on the data quality objectives, the compounds of concern, the media, and the exact sampling technique. The Contract Laboratory Program plans to issue a "Field Methodology Catalog" in the summer of 1987 that will contain field analytical techniques suitable for analyses of the samples collected by using the techniques in this section.

#### **13.1 WIPE SAMPLING**

##### **13.1.1 Scope and Purpose**

This guideline discusses the steps required for obtaining a wipe sample. Wipe samples may be used to document the presence of carcinogenic substances or other toxic materials. In addition, wipe sampling is commonly used to ascertain that site or equipment decontamination has been acceptably effective.

##### **13.1.2 Definitions**

###### **Site Manager (SM)**

The individual responsible for the successful completion of a work assignment within budget and schedule. This person is also referred to as the Site Project Manager or the Project Manager and is typically a contractor's employee (see Subsection 1.1).

###### **Wipe Sample**

A sample used to assess surface contamination. The terms "wipe sample," "swipe sample," and "smear sample" have all been used synonymously. For purposes of this section, the sample will be termed "wipe sample."

##### **13.1.3 Applicability**

This guideline is applicable when a sample of the substances on a surface is needed. Surfaces may include walls, floors, ceilings, desk tops, equipment, or other large objects that are potentially contaminated.

##### **13.1.4 Responsibilities**

The SM or designee is responsible for deciding when wipe sampling is needed.

Field personnel are responsible for performing the actual sampling, maintaining sample integrity, and preparing the proper chain-of-custody forms.

### 13.1.5 Records

Records of wipe sampling include completed chain-of-custody forms and appropriate entries in the field logbook. If the sample collected is to be analyzed using the National Contract Laboratory Program (CLP), then CLP forms must be completed as discussed in Section 5.

### 13.1.6 Procedures

Wipe sampling can be an integral part of the overall sampling program. Wipe sampling can help to provide a picture of contaminants that exist on the surface of drums, tanks, equipment, or buildings on a hazardous waste site or that exist in the homes of a populace at risk.

Wipe sampling consists of rubbing a moistened filter paper over a measured area of 100 cm<sup>2</sup> to 1 m<sup>2</sup>. The paper is then sent to the laboratory for analysis. The results are related back to the known area of the sample. A proper sampling procedure is essential to ensure a representative, uncontaminated sample.

#### 13.1.6.1 Equipment Required

The following equipment is needed for wipe sampling:

- Whatman 541 filter paper or equivalent, 15 cm
- Disposable, chemical-protective gloves
- Solvent to wet filter paper

#### 13.1.6.2 Wipe Sampling Steps

The steps involved in obtaining a wipe sample are listed below:

- Using a clean, impervious disposable glove, such as a surgeon's glove, remove a filter paper from the box. (Note: Although it is necessary to change the glove if it touches the surface being wiped, a new glove should be used for each sample to avoid cross contamination of samples. A new glove should always be used when collecting a new sample.)
- Moisten the filter with a collection medium selected to dissolve the contaminants of concern as specified in the sampling plan. Typically, organic-free water or the solvent used in analysis is used. The filter should be wet but not dripping.
- Thoroughly wipe approximately 1 m<sup>2</sup> of the area with the moistened filter. Using a 1 m<sup>2</sup> stencil will help in judging the size of the wipe area. If a different size area is wiped, record the change in the field logbook. If the surface is not flat, be sure to wipe any crevices or depressions.

- Without allowing the filter to contact any other surface, fold it with the exposed side in, and then fold it over to form a 90-degree angle in the center of the filter.
- Place the filter (angle first) into a clean glass jar, replace the top, seal the jar according to quality assurance requirements, and send the sample to the appropriate laboratory.
- Prepare a blank by moistening a filter with the collection medium. Place the blank in a separate jar, and submit it with the other samples.
- Document the sample collection in the field logbook and on appropriate forms, and ship samples per procedures listed in Section 6.

### 13.1.7 Region-Specific Variances

No region-specific variances have been identified; however, all future variances will be incorporated in subsequent revision to this compendium. Information on variances may become dated rapidly. Thus, users should contact the regional EPA RPM for full details on current regional practices and requirements.

### 13.1.8 Information Sources

EBASCO. "Dioxin Sampling." *REM III Program Guidelines*. Prepared for U.S. Environmental Protection Agency. 28 February 1986.

NUS Corporation. "Site-Specific Site Operations Plans." REM/FIT Contract.

## 13.2 HUMAN HABITATION SAMPLING

### 13.2.1 Scope and Purpose

This subsection provides general guidance for the planning, method selection, and implementation of sampling activities used to determine the potential for human exposure to contaminants that are present in residential environment.

### 13.2.2 Definitions

#### Human Habitation Areas

Any place people may spend extended periods of time, such as their homes or offices.

### 13.2.3 Applicability

This subsection discusses sampling techniques that are similar in collection methodology to other types of samples, such as environmental soil and water, but are biased to emphasize potential human exposure to contaminants moving into the residential environment.



## **APPENDIX C**

### **HEALTH AND SAFETY PLAN**

## HEALTH AND SAFETY PLAN

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| C3 | TVA Form 9179, Claim of Disability (Job-Related)                                 |
| C4 | TVA Form 255, TVA Vehicle Accident, Theft, or Fire                               |
| C5 | Form SR-13, Alabama Department of Public Safety -<br>Involving a Private Vehicle |
| C6 | First Aid Instructions for TVA Employees   |

## 1.0

### GENERAL

The overall risks associated with this operation include danger to those being in (or near) the area where low concentrations of explosive materials are handled, working with and near hot, heavy metal objects, and handling chemicals used in sampling procedures to wash out metal objects. Fall and trip hazards exist as related to elevated work platforms (scaffolding). There is a moderate risk of back injury/strains from lifting heavy metal objects to be sampled. A moderate risk exists from pinches, scrapes, cuts, and abrasions. A moderate risk exists from hazards associated with heat stress (heat cramps, heat exhaustion, heat stroke). Inhalation from dust hazards is low. A low risk exists from snake bites, spider bites, and scorpion stings.

Risks will be minimized by following carefully designed safe work practice procedures and standing operating procedures. Handling of explosive materials will be by DZB, the site contractor. DZB will also be responsible for loading/unloading railcars, operating the decontamination process equipment, and using the manipulators to move heavy metal parts for sampling.

A functional chart depicting part of HWAAP Safety Organization is included as Figure C-1. TVA site manager will report to HWAAP Commanding Officer with indirect reporting to HWAAP Safety Director. All TVA field personnel will be under functional control of TVA's site manager.

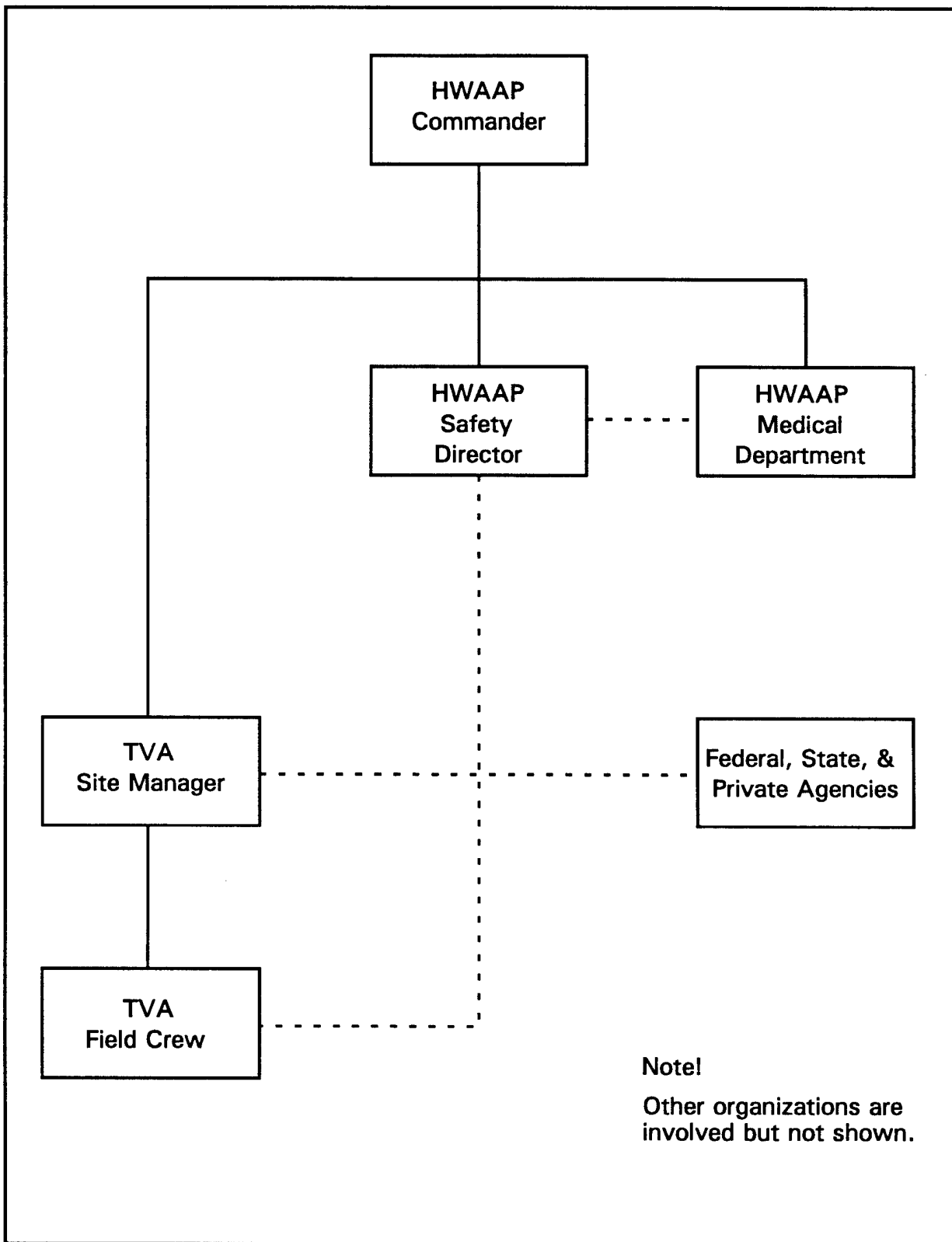


Figure C.1 HWAAP Safety Organization Functional Chart (Partial)

## 2.0

### KEY PERSONNEL

<u>Position</u>	<u>Name</u>	<u>Phone No.</u>
<b>Hawthorne Army Ammunition Plant:</b>		
Admin Contracts Officer	Tiny Cardenas	(702) 945-7341
Project Officer	Herman Milsap	(702) 945-7317
Project Engineer	Louie Delamonica	(702) 945-7354
Health & Safety Officer		(702) 945-7020
<b>Tennessee Valley Authority:</b>		
Project Manager	Rick Almond	(205) 386-3030
Site Manager	James R. Watson	(702) 945-
<b>U.S. Army Environmental Center:</b>		
Project Officer	Erik Hangeland	(410) 671-1556
Safety & Environmental	William Houser	(410) 671-4811
Services Branch	Marty Stultz	(410) 671-1568
<b>U.S. Army Environmental Hygiene Agency:</b>		
Project Officer	CPT J Schuliger	(410) 671-3500
<b>Site Contractor (Day &amp; Zimmerman/Basil Corporation)</b>		
Project Manager	Jim Reese	(702) 945-7658

## 3.0

### MAPS OF WORK AREAS

Figure 1 through Figure 4 (Section 1) indicate the areas within WADF and HWAAP in which TVA personnel will be working. Other areas may require access as the project progresses. Where as TVA personnel may be required to work in other areas not indicated, most of the work will be within these boundaries. Visits to other parts of HWAAP may require a guide or escort.

## 4.0

### PERSONNEL PROTECTIVE CLOTHING AND EQUIPMENT

### 4.1

#### General

As a minimum, the 1993 Hawthorne Army Ammunition Plant Accident Prevention Program manual will serve as TVA's personnel protective clothing and equipment guide. All protective clothing and equipment must meet the minimum standards prescribed by ANSI, OSHA, TVA, and/or MIL Specs.

#### **4.2**      **Responsibilities**

The TVA site manager will have overall responsibility to ensure that all TVA personnel utilize the protective clothing and equipment prescribed of a particular job. Each individual shall be familiar with the necessary clothing and equipment to do their job and shall keep all items serviceable at all times.

Any faulty equipment shall be reported to the appropriate DZB personnel for repairs and or replacement. (TVA personnel shall not attempt to repair any equipment other than that belonging to TVA).

#### **4.3**      **Protective Clothing and Equipment**

When required, all personnel will wear non-sparking conductive safety footwear when working on conductive flooring, matting, runners, where conductive materials are required by DZB SOP, or other standards and regulations. Safety shoes will be furnished by TVA.

The HWAAP Warehouse Branch will provide gloves, safety goggles, coveralls, hard hats, and if required, respiratory protection equipment. All protective clothing and equipment will be stored in TVA's van or office trailer parked adjacent to Building 117-15. Conductive footwear will not be worn as street shoes and will be left at the work place at the end of each shift.

All items received from the Warehouse Branch will be accounted for and returned to HWAAP prior to TVA's departure from Hawthorne Army Ammunition Plant at the completion of the test plan project.

#### **4.4**      **Prescription Safety Glasses**

TVA personnel requiring prescription glasses shall provide their own prescription safety glasses and wear goggles where appropriate at HWAAP. Contact lenses will not be worn on site.

#### **4.5**      **Hard Hats**

TVA personnel will be issued hard hats from HWAAP Warehouse branch upon arrival. The hard hats will be worn when entering a designated hard hat area. The flashing furnace area is not considered a hard hat area and soft caps will be allowed during normal duty at that site. The hard hats will be maintained in the working area should any person be required to work temporarily at another site.

## **5.0 TRAINING**

### **5.1 General**

TVA personnel involved in field sampling at HWAAP and laboratory analysis at Muscle Shoals, Alabama, shall be adequately trained for handling explosives. USADACS safety personnel conducted a training course for TVA personnel at Muscle Shoals, Alabama, prior to departure for HWAAP. All sampling procedures, chamber loading techniques, and contaminated test items handling procedures will be field tested (at HWAAP) prior to execution of the proposed test plan.

### **5.2 Laboratory Plan/Protocol**

All TVA laboratory personnel will use the TVA approved laboratory plan for handling samples for analysis, based on established practices for hazardous and toxic materials and the specific requirements of the explosive being tested. The Laboratory Protocol (Appendix A) and Methods and Procedures (Appendix B) of this plan covers all laboratory analysis and field sampling procedures.

### **5.3 On Site Training**

TVA personnel shall receive site specific training and orientation as deemed necessary by HWAAP/DZB in conjunction with in-processing and setup upon arrival at HWAAP.

All pertinent subjects shall be covered to include but not necessarily limited to:

- 1) Site security
- 2) Site safety precautions/regulations
- 3) Site warning signals
- 4) Site orientation and facility locations
- 5) Site specific rules and regulations
- 6) Severe weather warnings/conditions/actions
- 7) General briefing on local customs/general conditions
- 8) Emergency response actions

## **6.0 MEDICAL PROTOCOL**

### **6.1 Key Medical Personnel**

Key medical personnel necessary to support any emergencies will be the emergency medical technicians (EMTs) provided by the HWAAP fire department (Phone 7911). In the event of an accident or illness beyond the scope of the EMTs, Mount Grant General Hospital, First and A Streets, (Phone 945-2461), in



the city of Hawthorne, Nevada, would be used. Mount Grant's medical staff would make the arrangements necessary to air evacuate the patient to either Washoe Medical Hospital, 77 Pringle Way, (Phone 328-4100) or St. Mary's, Regional Medical Center, 235 West Sixth Street, (Phone 789-3188) in Reno, Nevada, should the medical situation dictate. Figure C-2 illustrates the highway route from Hawthorne to Reno, Nevada. The location of both St. Mary's and Washoe Medical Hospitals are shown in Figure C-3.

TVA's site manager will coordinate all field activities (through HWAAP's staff) with the emergency medical facilities at HWAAP. This will ensure appropriate medical coverage and support is always available in the event a medical emergency occurs.

## **6.2      Emergency Response Equipment**

The Hawthorne Army Ammunition Plant fire department will provide emergency response equipment and personnel within the HWAAP boundary. All TVA personnel will adhere to the HWAAP/DZB SOP for emergency response. Emergency response actions and responsibilities will be included in the initial briefing received by TVA upon arrival at HWAAP.

TVA will provide Material Safety Data Sheets (MSDS), or equivalent information, to include health hazard information and chemical and physical properties, of all decon solutions, preservatives, and calibration gases brought on site, to the HWAAP fire department. The information will include a list of all chemicals, quantities, and storage locations. TVA will also maintain the information in the office trailer at the job site.

This information will be provided at any time additional chemicals are purchased and delivered to HWAAP. All listings will be kept current.

## **6.3      First Aid Procedures**

All TVA employees are familiar with first aid procedures. A first aid instructional manual for TVA employees is part of this manual. TVA field sampling personnel have been trained in CPR techniques and will undergo a refresher course prior to departure for HWAAP.

TVA's site manager will coordinate any additional training or first aid-related subjects with the safety office at HWAAP. This will be handled with assistance from the Administrative Contract's Officer at HWAAP.

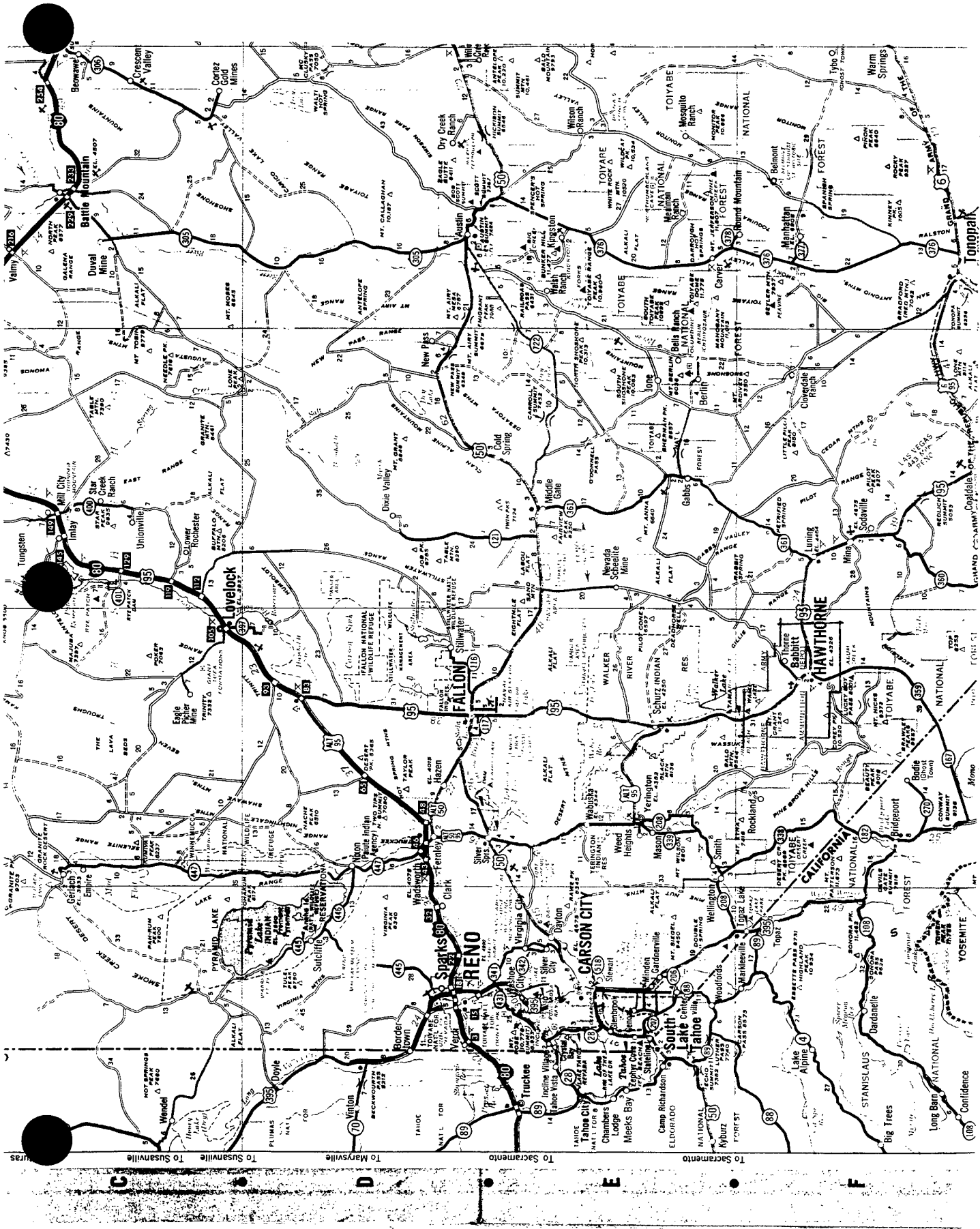


Figure C-2 Route from HWAAP to Reno, Nevada

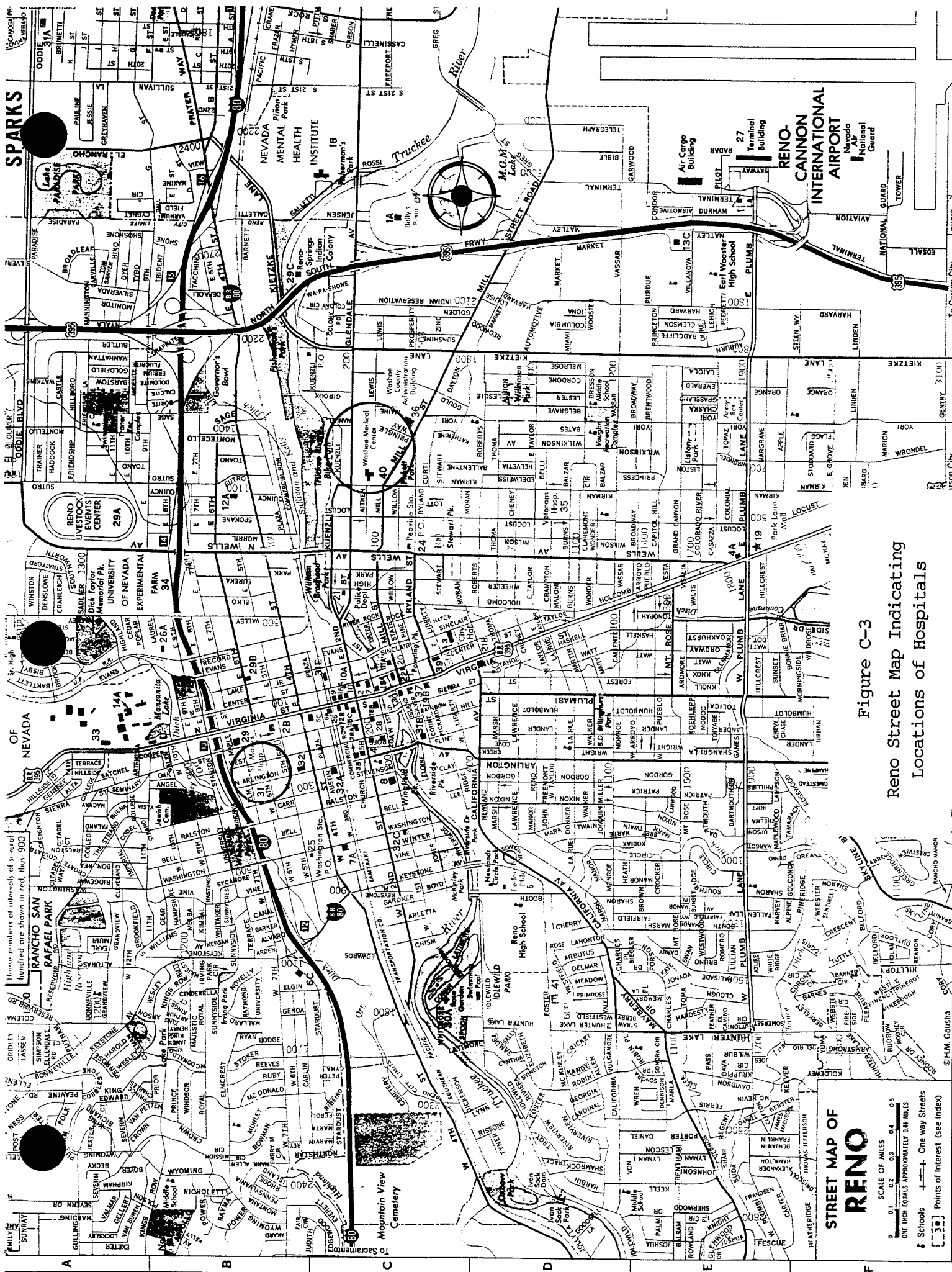


Figure C-3  
Reno Street Map Indicating  
Locations of Hospitals

**STREET MAP OF  
RENO**

SCALE OF MILES  
0 0.1 0.2 0.3 0.5  
ONE INCH EQUALS APPROXIMATELY 0.4 MILES

• Schools    + One way Streets  
[ 3 ] Points of Interest (see index)

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W 3RD ST  
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5-14660

## 7.0

### PERSONAL HYGIENE

Eating, drinking, chewing, and smoking will be prohibited within the working area. Only areas designated for that purpose will be used. Personnel will be required to wash their hands prior to eating and at any time following the use of chemicals and contaminated explosive items and at any time contamination is expected.

Smoking materials such as cigarettes, cigars, pipes, matches, lighters, etc., will not be permitted in the work area. All such items will be left in the designated areas and not carried on the person.

## 8.0

### PERSONNEL REQUIREMENTS

The TVA field sampling team will consist of the following:

- 1 - Site Manager
- 1 - Chemist
- 1 - Chemical Lab Analyst

The TVA laboratory analysis team will consist of the following:

- 1 - Laboratory Manager
- 1 - Quality Assurance Officer
  - Research Chemist - As required
  - Chemical Laboratory Analysts - As required

The TVA project management team will consist of the following:

- 1 - Project Manager
- 1 - Chemical Engineer
- 1 - Project Engineer
- 1 - Cost/Schedule Engineer
  - Project Engineers - As required
  - Computer Analysts - As required
  - Others - As required

## 9.0

### VISITOR POLICY

All visitors to HWAAP will be required to sign in through the HWAAP security office to obtain a visitor's badge and vehicle pass. All visitors to the Hot Gas Decontamination for Explosives Project are required to sign in with the TVA site manager to record the visitors and purpose of the visit. A daily log will be maintained by the TVA site manager of all activities during the project.

## **10.0 SUPPORT FROM HWAAP AND DZB**

HWAAP maintains qualified and trained Emergency Medical Technicians (EMTs), fire fighting personnel, environmental officers, and safety officers on site. EMTs and fire fighting personnel are on duty 24 hours per day. HWAAP EMTs will provide rapid response to emergencies and transportation to designated local hospitals.

HWAAP's Administrative Contracts Officer will serve as TVA's point of contact with DZB as well as the initial contact for assistance for other HWAAP offices (i.e., medical, environmental, safety, and fire).

## **11.0 EMERGENCY CONTINGENCY PLAN/ACCIDENT REPORTING**

### **11.1 Emergencies**

Should events, incidents, or accidents occur beyond the scope of this plan, TVA personnel will take direction from HWAAP/DZB to evacuate the plant site to a safe location as designated. Return to normal duties will follow an "all clear" notification from HWAAP.

### **11.2 Accidents and Reports**

Accidents resulting in a fatality, lost-time injury or illness, hospitalization of five (5) or more personnel, or property damage to government or contractor property (which occurred during the performance of the contract) equal to or exceeding \$2,000.00 must be telephonically reported to the U. S. Army Environmental Center (USAEC), SFIM-AEC-TSS, phone number (410) 671-4811, as soon as possible, but not later than two (2) hours after occurrence and reported in writing within five (5) days of occurrence on DA Form 285 (enclosed). All other accidents/incidents must be telephonically reported to USAEC, SFMIM-AEC-TSS, phone number (410) 671-4811, within eight (8) hours of occurrence.

An accident report form (DA Form 285, January 92), with instructions, is included at the end of this appendix. Additional forms may be obtained from the HWAAP Safety Office.

Accidents will also be reported using TVA protocol and procedure through TVA's chain of command and using the appropriate TVA forms and instructions. Accident reporting forms are included at the end of this appendix.

- Form CA-1 Federal Employee's Notice of Injury (Job Related)
- TVA 9179 Claim of Disability (Job Related)
- TVA 255 TVA Report of Vehicle Accident, Theft, or Fire
- SR 13 Alabama Department of Public Safety (Private Vehicle)

## **12.0**      **SECURITY**

### **12.1**      **General**

The facilities and equipment at Hawthorne Army Ammunition Plant, Hawthorne, Nevada, as well as the plans, methodologies, and literature contained in this document, are "unclassified" concerning national security. Security, as it relates to personnel-allowed access to buildings, will be imposed to control personnel at the test site. HWAAP security personnel will control the public's access to HWAAP and WADF.

### **12.2**      **Site Control Measures**

HWAAP security will control access to all sites within the plant area. Portable barricades in conjunction with existing gates may be used to control access when necessary.

### **12.3**      **Badges**

TVA personnel will be issued permanent non-escorted identification badges upon arrival at HWAAP. These badges will be issued in accordance with normal and routine HWAAP security procedures and all badges will be surrendered to HWAAP security department upon completion of this field testing period.

### **12.4**      **Vehicles**

TVA's vehicles, whether U.S. Government vehicles or rental units, will be issued permanent passes upon arrival at HWAAP according to normal security SOP. TVA vehicles will be subject to all governing rules and regulation enforceable at the time the test plan is executed. Passes will be surrendered at the completion of the field testing period.

### **12.5**      **Cameras**

All TVA personnel will be required to register cameras with HWAAP's security department upon arrival. A camera pass will be required for each camera carried on site.

### **12.6**      **Access**

TVA's site manager will control access to the TVA test facilities (vans and trailers) in coordination and cooperation with HWAAP/DZB. However, security for this operation will be under the direct supervision of HWAAP security personnel in strict compliance with all enforced procedures and regulations.

12.7

Firearms

TVA personnel will not be allowed to carry firearms on HWAAP.

13.0

SIGNATURES

The undersigned have read this Health and Safety Plan, understand its contents, and will comply with all provisions contained herein. (This record will be kept on site.)

SIGNATURE

DATE

SIGNATURE

DATE

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**APPENDIX C1**

**DA FORM 285 (JAN 92)**

**U. S. ARMY ACCIDENT REPORT**



# U.S. ARMY ACCIDENT REPORT Instructions

**General.** The unit having the accident must investigate it and complete this report. Complete the shaded portions **only** for: Military on-duty, on-fatal accidents; and military on-duty accidents resulting in less than 20 lost workdays. Accidents involving 20 or more lost workdays and/or total property damage of \$2,000 or more will require completion of the entire report. Type or legibly print the report. Items may be continued on a blank sheet of paper and attached to the report. Items listed below are keyed to the block numbers of DA Form 285, May 91. Items not listed here are self-explanatory. Specific questions concerning this form should be referred to the local safety office.

## SECTION A - Accident Information

**Note:** This section should be completed for the initial report and for any changes to a previously submitted report.

1. Check "INITIAL" if this is the first report on the accident. Check "CHANGE" if this report is a change to a previously submitted report of the accident.
2. Enter the 6-digit Unit Identification Code (UIC) for the unit responsible for the accident (e.g., WXXXXX).
3. Provide military unit information for the unit listed in Block 2.
  - a. Full military address (e.g., C Troop, 17 Cavalry, Ft. Bragg, NC 12345-6789).
  - b. Provide the unit branch (e.g., Armor, Infantry, Transportation).
4. Enter the year, month, and day of the accident (e.g., 90 11 07 (7 November 1990)).
5. Enter the military time the accident occurred (e.g., 0815, 2300).

Check either item a or b, depending on the location of the accident.

If item a is checked, state name of post or installation (e.g., Ft. Bragg, NC; Federal Center, Atlanta, GA; Ft. Hood, TX; Shaw AFB, SC).

9. Check item a if accident occurred in a theater of hostile fire or enemy action, but not as a result of such fire/action. This includes direct preparation for combat, actual combat, or redeployment from a combat theater.
10. Check "Yes" if explosives (C-4, TNT), ammunition, or pyrotechnics were involved and explain in Block 63 its involvement and specify the National Stock Number (NSN).
11. Give enough detail to find the exact location of the accident (e.g., building number, street or highway name, state and/or country). Also state the type of location (e.g., road intersection, tank trail, family housing, firing range).

## SECTION B - Personnel Information

**Note:** Complete this section for each individual involved and/or injured in the accident. "Involved" means any person who was injured, or who took actions, or made decisions which caused or contributed to the accident. If more than one person was involved, enter information on one person on the initial form and complete only Sections A and B on additional forms for others. Staple all forms together.

16. Enter individual's rank/grade (e.g., E5/SGT, O3/CPT, GS-11, WG-8). Complete for all Government personnel.
17. Enter individual's full MOS/Job Series (e.g., 54E20, 11B40, GS-301).
18. Provide individual's full **Military** address for Government personnel. If this address is not same as that in Block 3a, provide the unit.

21. State how many continuous hours without sleep this individual was on-duty prior to the accident.

22. Indicate how many hours of continuous sleep this individual had in the past 24 hours.

23. State the estimated number of days this individual will be away from work (totally unable to perform any work but retain quarters). Does not include days hospitalized.

24. State the estimated (or actual) number of days this individual is hospitalized (inpatient/somnolent) receiving treatment. Days hospitalized for "observation only" are not reported.

25. State the estimated number of days this individual will not be able to perform his or her regular duties (light duty, profile).

26. Check appropriate block. If more than one applies, check the most severe.

28. For this individual's "most severe injury", check the appropriate block(s) (no more than 3) that indicate the cause of the injury.

29. Number the body part(s) most seriously injured (no more than 3) in their order of priority (the most serious first). Be as specific as possible.

30. For each body part numbered in block 29, place a corresponding number to indicate the type of injury received (select only the most serious).

31. Check the appropriate block that best describes the individual's action at the time of the accident. If Block 31gg is checked, complete Blocks 76 and 77 of Section H, as indicated by these instructions.

32. Provide a short but detailed explanation of the item checked in Block 31.

**Note:** For this report, the following definitions apply:

**Tactical Training** - Training in a field environment that uses or develops combat or combat support skills.

**Field Exercise and Tactical Training** - This begins when the individual reports to his or her primary duty location for movement to the field site and ends when he or she arrives back at the primary duty location from the field.

33. Check "Yes" if activity listed in Block 31 was part of a field exercise. State name of exercise if it has a name (e.g., Team Spirit, Reforger).

42. If vision enhancement device(s) were used, specify type and model numbers, and whether they caused the accident (e.g., Night Vision Goggles, AN-PV55A).

43. Provide standard or reference (Soldier's Manual, AR TM, etc.), if it exists, that covers performance of the activity identified in Block 31.

46. Provide a simple explanation of the mistake(s) or how the activity or task was performed incorrectly (e.g., SGT Smith improperly backed his M915 truck without a ground guide).

47. In your opinion, why was the mistake made or the activity performed incorrectly? Check the most important reason.

51. Check the block corresponding to the piece of equipment associated with the person in Block 12 (e.g., SGT Adams was driving the "at-fault" HMMWV, his name will be in Block 12, and his vehicle will be item a in Section C below).

## SECTION C - Property/Material Involved

Complete Blocks 52-59 on each piece of property or item of equipment involved in the accident (whether damaged or not). Include Army and non-Army, as well as equipment whose use or misuse contributed to the accident. Include up to 3 items of equipment on the initial form. Use additional blank sheets of paper for other equipment if necessary, continuing letter sequence (e.g., A, B, C, D, and E).

52. Type of equipment (e.g., sedan, truck, generator).

53. Full military equipment model number or civilian make (e.g., M109A2, M60A2, Ford Taurus, M16 Rifle).

55. Estimated cost of damage (ECOD) or actual cost of damage (ACOD) for each piece of property, which includes costs of parts and labor.

57. Indicate if this specific item was being towed at the time of the accident.

58. If Block 57 is "yes", indicate which item was doing the towing.

60. Complete for each component or part whose failure or malfunction contributed to the accident. Include the EIR/QDR number in Block 60e.

61. Indicate how and why each component or part failed or malfunctioned by selecting from the lists provided and entering the appropriate number in the blocks provided.

## SECTION D - Environmental Conditions Involved

62. Check the environmental conditions present at the time of the accident (no more than 3) by checking appropriate blocks, whether contributing to the accident or not. Also check whether they caused or contributed to the accident.

## SECTION E - Accident Description/Narrative

63. Fully describe the sequence of events that lead up to and caused the accident. Explain how and why the accident occurred. Also include information required from Blocks 10 and 47.

## SECTION F - Corrective Action and Command Review

**Note:** The level of command review (Company, Battalion, Division, etc.) is determined by either the major Army command (MACOM) or installation policy.

65. Fully describe all actions taken, planned, or recommended to eliminate the causes of this accident. Actions should be identified as appropriate at unit level, and all the way up to HQDA level.

## SECTION G - SAFETY OFFICE USE ONLY

71. MACOM responsible for this accident (FORSCOM, TRADOC, etc.).

## SECTION H - Special Interest/Supplemental Information

This section is for use by the U.S. Army Safety Center, MACOMs, or interested safety offices to obtain additional "Special Interest/Supplemental Information" on this accident as needed (e.g., M tank fires, tactical parachute accidents, etc.). Blocks 76 and 77 have been designated for collection of supplemental information on parachuting accidents.

Blocks 76 and 77. If Block 31gg was checked, provide the following supplemental information for each individual:

- a. Name of jumper.
- b. Jumper height.
- c. Jumper weight.
- d. Type of jump (static line, non-tactical; static line, mass technical; freefall non-tactical; freefall, tactical).
- e. Type of parachute and model.
- f. Jumper's equipment (list).
- g. Weight of equipment.
- h. Wind direction and speed at
  - (1) Jump height.
  - (2) Drop zone.
- i. Jump altitude.
- j. Jumper's position in stick and door exit.
- k. Time pre-jump conducted.
- l. Date of last jump and type of jump.
- m. Number of previous jumps.
- n. Date graduated from basic airborne training (year and month).
- o. Type of aircraft.
- p. Accident cause(s): Improper exit, static line injury, broken static line, parachute malfunction, entanglement, lost or stolen air, oscillation, unstable position, dragged on DZ, free landing, drop zone hazard (specify), or other.



# SECTION B - PERSONNEL INFORMATION (Continued)

31. Person's activities at time of accident (Check one and explain in Block 32.)

a. Soldering	j. Test/Study/Experiments	s. Fabricating	33. Hobbies
b. Combat Soldiering	k. Educational	t. Handling Material/Passengers	34. Passenger
c. Physical Training	l. Information and Arts	u. Junior/Novice/ Housekeeping/ Grounds Keeping	35. Human movement
d. Weapons Firing	m. Food and Drug Inspection	v. Food/Drink Preparations	36. Horseplay
e. Engineering or Construction	n. Laundry/Dry Cleaning Services	w. Supervisory	37. Bystanding/spectating
f. Communications	o. Pest/Plant Control	x. Office	38. Personal Hygiene/Food/Drink Consumption/Sleeping
g. Security/Law Enforcement	p. Operating Vehicle or Vessel	y. Courtesy/Advisory	39. Parachuting (See instructions)
h. Fire Fighting	q. Handling Animal	z. Sports	
i. Patient Care (People/Animals)	r. Maintenance/Repair/Service		

32. SPECIFIC DESCRIPTION OF ACTIVITY/TASK

33. ON FIELD EXERCISE (Check one)  
☐ a. Yes (If YES, specify name of exercise.)  
☐ b. No

34. ACTIVITY PART OF TACTICAL TRAINING? (Check one)  
☐ a. Yes  
☐ b. No

35. Type of training facility being used (Check one)

a. Garrison	d. NTC	g. Std. range facility/ live fire
b. Local training area	e. JRTC	h. Other (Specify)
c. Main training area	f. CMTC	

36. Type of training participating in at the time of accident (Check/Specify)

a. School (Specify)	
b. Unit → (1) Platoon (2) Crew (3) Individual	
c. On-the-job training	d. Other (Specify)

37. Last time individual received training prior to accident on activity specified in block 31? (Check one)

a. 0 - 3 months	u. 1 - 2 years
b. 3 - 6 months	v. More than 2 years
c. 6 - 9 months	w. Never
d. 9 - 12 months	x. Not applicable

38. Required protective equipment

CHECK APPROPRIATE BLOCK(S)	AVAILABLE?		USED?		N/A
	YES	NO	YES	NO	
a. Seat belt					
b. Helmet					
c. Goggles/glasses					
d. Gloves					
e. Ear plugs					
f. Other (Specify)					

39. INDIVIDUAL LICENSED TO OPERATE VEHICLE/EQUIPMENT? (Check one)

☐ a. Yes ☐ b. No ☐ c. N/A

40. DID ALCOHOL CAUSE/CONTRIBUTE TO THIS ACCIDENT? (Check one)

☐ a. Yes ☐ b. No ☐ c. Unknown

41. If drugs caused/ contributed to this accident, check appropriate block.

a. Prescription
b. Illegal
c. Over-the-counter
d. None

42. Were vision enhancement devices being used? (Check appropriate block.)

a. Yes (Specify type/model in c and d.)	
b. No	
c. TYPE	d. MODEL

43. Standard/Reference covering activity/task

a. Soldier's Manual (Task No.)	
b. CTT (Task No.)	
c. AR/TM/FM (Specify)	
d. SOP	e. None (Go to block 45.)

44. WAS ACTIVITY/TASK PERFORMED IAW STANDARD/REFERENCE? (Check one)

☐ a. Yes ☐ b. No (If NO, complete blocks 46-47)

45. DID INDIVIDUAL MAKE A MISTAKE? (Check one)

☐ a. Yes (If YES, complete blocks 46-47) ☐ b. No

46. What was the mistake? How was the activity/task performed incorrectly? (Explain below.)

47. Why was mistake made/activity performed incorrectly? (Check the most important reason and specify in Block 63.)

a. Inadequate school training (content/amount)	i. In a hurry	k. Inadequate services
b. Inadequate unit training (content/amount)	g. Poor/bad attitude	l. Improper equipment design
c. Inadequate on-the-job training (content/amount)	h. Lack of resources	m. Inadequate written procedures (AR, TM, SOP)
d. Fear/ excitement	j. Effects of alcohol/drugs	n. Improper supervision
e. Overconfident in one's/other's abilities	l. Inadequate facilities	o. Other (Specify in narrative)

# SECTION B - PERSONNEL INFORMATION (Continued)

48. Time licensed on this vehicle (Check one)		49. Total AMV driving mileage (Check one)		50. Total time in unit (Check one)	
a. Less than one year		a. Less than 1,000 miles		a. Less than 6 months	
b. One to two years		b. 1,000 - 5,000 miles		b. 6 months - 1 year	
c. Over two years		c. 5,000 - 10,000 miles		c. Over one year	
d. Unlicensed		d. Over 10,000 miles			

51. WHICH ITEM FROM SECTION C APPLIES TO THE INDIVIDUAL NAMED IN BLOCK 12? (This is needed in order to relate the person in block 12 to the equipment/vehicle below.)

☐ Item A    ☐ Item B    ☐ Item C    ☐ OTHER (Specify)

# SECTION C - PROPERTY/MATERIAL INVOLVED (Whether Damaged or Not)

	ITEM A	ITEM B	ITEM C
52. Type of item			
53. Model number			
54. Ownership (DOD CA POV Unit Person)			
55. Dollar cost of damage			
56. Rollover protection system installed?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA
57. Was this item being towed?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> NA
58. If towed, enter letter for item doing towing			
59. Types of collision codes (Pick up to three from list below and enter in blocks.) (in sequence)			

## Types of Collisions

- |   |  |
|---|--|
| 1 - Going forward and collided with moving vehicle      | 7 - Ran off the road                             |
| 2 - Going forward and collided with parked vehicle      | 8 - Jackknifed                                   |
| 3 - Collision while backing                             | 9 - Going forward and rear-ended moving vehicle  |
| 4 - Collision with pedestrian                           | 10 - Going forward and rear-ended parked vehicle |
| 5 - Collision with object other than vehicle/pedestrian | 11 - Collision while turning                     |
| 6 - Overturned  | 12 - Other (Specify)                             |

## 60. Component/Part that Failed/Malfunctioned (Complete this section if a material failure/malfunction caused/contributed to the accident.)

	ITEM A	ITEM B	ITEM C
a. National Stock Number			
b. Part Number			
c. Describe Part			
d. Manufacturer's Identification Code			
e. EIR/QDR Number			

61. How/Why Part Malfunctioned (Select code from "How" list below and enter in first block; select code from "Why" list and enter in second block.)	HOW	WHY	HOW	WHY	HOW	WHY

## How Part Failed/Malfunctioned Codes

- |                                |                               |
|--------------------------------|-------------------------------|
| 1 - Overheated/burned/melted   | 9 - Twisted/torqued           |
| 2 - Froze (temperature)        | 10 - Compressed/hit/punctured |
| 3 - Obstructed/pinched/clogged | 11 - Bent/warped              |
| 4 - Vibrated                   | 12 - Sheared/cut              |
| 5 - Rubbed/worn/trayed         | 13 - Decayed/decomposed       |
| 6 - Corroded/rusted/pitted     | 14 - Electric current action  |
| 7 - Overpressured/burst        | 15 - Unknown/Other            |
| 8 - Pulled/stretched           | Blank - Not reported          |

## Why Part Failed/Malfunctioned Codes

- 1 - Improper equipment design
- 2 - Inadequate maintenance
- 3 - Inadequate manufacture of equipment
- 4 - Inadequate written procedures (AR, TM, SOP)
- 5 - Improper supervision
- 6 - Unknown
- 7 - Other (Specify in narrative)

# SECTION D - ENVIRONMENTAL CONDITIONS INVOLVED

62. Environmental conditions. (Check environmental conditions present and indicate if condition caused/contributed to the accident.)

PRESENT	CAUSED/ CONTRIBUTED	CONDITION	PRESENT	CAUSED/ CONTRIBUTED	CONDITION
		a. Clear/dry, visibility unlimited			k. Wind gust/turbulence
		b. Bright, glare			l. Vibrate, shimmy, sway, shake
		c. Dark, dim			m. Radiation (aser, sunlight)
		d. Fog, condensation, frost			n. Holes, rocky, rough, rutted, uneven
		e. Mist, rain, sleet, hail			o. Inclined/sloped
		f. Snow, ice			p. Slippery (not due to precipitation)
		g. Dust, fumes, gasses, smoke, vapors			q. Air pressure (dense, decompression, altitude, hypoxia)
		h. Noise, bang, static			r. Lightning, static electricity, ground
		i. Temperature/humidity (cold, heat)			s. OTHER (Specify)
		j. Storm, hurricane, tornado			

# SECTION E - ACCIDENT DESCRIPTION/NARRATIVE (From blocks 10, 47)

63. GIVE THE SEQUENCE OF EVENTS THAT AMPLIFY/EXPLAIN WHAT HAPPENED, LEADING UP TO AND INCLUDING THE ACCIDENT. (Explain why accident happened.)

64a. PRINTED/TYPED NAME OF PERSON COMPLETING THIS REPORT

64b. RANK

64c. TITLE

64d. SIGNATURE

64e. DATE OF SIGNATURE  
(YY/MM/DD)

64f. TELEPHONE NO.

# SECTION F - CORRECTIVE ACTION AND COMMAND REVIEW

65. DESCRIBE THE ACTIONS TAKEN, PLANNED, OR RECOMMENDED TO ELIMINATE THE CAUSE(S) OF THIS ACCIDENT (from Unit level up to HQDA).

66a. PRINTED/TYPED NAME OF COMMANDER

66b. RANK

66c. SIGNATURE

66d. DATE OF SIGNATURE  
(YY/MM/DD)

66e. TELEPHONE NO.

a. TYPED NAME

b. SIGNATURE

c. TITLE

d. RANK / DATE

67

68

# SECTION G - SAFETY OFFICE USE ONLY

70. LOCAL REPORT NO

71. MACOM

72. Accident type (Check choice)

a. Army Motor Vehicle

n. Other Army Vehicle

o. Personal Injury - Other

b. Army Combat Vehicle

i. Fire

p. Property Damage - Other

c. Army Operated Vehicle

j. Chemical Agent

q. POV - On Official Business

d. POV - Not on Official Business

k. Explosive

r. Space

e. Marine Diving

l. Missile

s. Commercial Carrier/Transportation

f. Marine Underway

m. Radiation

g. Marine Not Underway

n. Nuclear

73. NAME OF SAFETY POINT OF CONTACT (POC)

74. PHONE NO OF SAFETY OFFICE POC  
(AUTOVON, Commercial, Etc.)

75. DATE REPORT COMPLETED BY  
SAFETY OFFICE (YY/MM/DD)

# SECTION H - SPECIAL INTEREST AND/OR SUPPLEMENTAL INFORMATION

76

77

78

79

**APPENDIX C2**

**U. S. DEPARTMENT OF LABOR FORM CA-1 (NOV. 89)**

**FEDERAL EMPLOYEE'S NOTICE OF TRAUMATIC INJURY AND CLAIM**

**FOR CONTINUATION OF PAY/COMPENSATION**

Federal Employee's Notice of  
Traumatic Injury and Claim for  
Continuation of Pay/Compensation

U.S. Department of Labor  
Employment Standards Administration  
Office of Workers' Compensation Programs



Employee: Please complete all boxes 1 - 15 below. Do not complete shaded areas.

Witness: Complete bottom section 16.

Employing Agency (Supervisor or Compensation Specialist): Complete shaded boxes a, b, and c.

Employee Data

1. Name of employee (Last, First, Middle)				2. Social Security Number	
3. Date of birth Mo. Day Yr. 	4. Sex <input type="checkbox"/> Male <input type="checkbox"/> Female	5. Home telephone (      )		6. Grade as of date of injury Level Step	
7. Employee's home mailing address (Include city, state, and zip code)				8. Dependents	
				<input type="checkbox"/> Wife, Husband <input type="checkbox"/> Children under 18 years <input type="checkbox"/> Other	

Description of Injury

9. Place where injury occurred (e.g. 2nd floor, Main Post Office Bldg., 12th & Pine)

10. Date injury occurred Mo. Day Yr. 	Time : <input type="checkbox"/> a.m. <input type="checkbox"/> p.m.	11. Date of this notice Mo. Day Yr. 	12. Employee's occupation
13. Cause of injury (Describe what happened and why)			

14. Nature of injury (Identify both the injury and the part of body, e.g., fracture of left leg)	a. Occupation code	
	b. Type code	c. Source code
	OWCP Use - NOI Code	

Employee Signature

15. I certify, under penalty of law, that the injury described above was sustained in performance of duty as an employee of the United States Government and that it was not caused by my willful misconduct, intent to injure myself or another person, nor by my intoxication. I hereby claim medical treatment, if needed, and the following, as checked below, while disabled for work:

- ☐ b. Continuation of regular pay (COP) not to exceed 45 days and compensation for wage loss if disability for work continues beyond 45 days. If my claim is denied, I understand that the continuation of my regular pay shall be charged to sick or annual leave, or be deemed an overpayment within the meaning of 5 USC 5584.
- ☐ a. Sick and/or Annual Leave

Signature of employee or person acting on his/her behalf \_\_\_\_\_

Any person who knowingly makes any false statement, misrepresentation, concealment of fact or any other act of fraud to obtain compensation as provided by the FECA or who knowingly accepts compensation to which that person is not entitled is subject to civil or administrative remedies as well as felony criminal prosecution and may, under appropriate criminal provisions, be punished by a fine or imprisonment or both.

Have your supervisor complete the receipt attached to this form and return it to you for your records.

End of Employee Report

Witness
16. Statement of witness (Describe what you saw, heard, or know about this injury)

Name of witness	Signature of witness	Date signed
Address	City	State Zip Code



**Official Supervisor's Report: Please complete information requested below:**

**Supervisor's Report**

17. Agency name and address of reporting office (Include city, state, and zip code)		OWCP Agency Code
TENNESSEE VALLEY AUTHORITY		
Workers' Compensation and Rehabilitation Department		OSHA Site Code
10 E. 11th. Street, EB 7A-C		
CHATTANOOGA, TN 37402-2801		Zip Code

18. Employee's duty station (Street address and zip code) Zip Code

19. Regular work hours	From: <input type="checkbox"/> a.m. <input type="checkbox"/> p.m.	To: <input type="checkbox"/> a.m. <input type="checkbox"/> p.m.	20. Regular work schedule	<input type="checkbox"/> Sun. <input type="checkbox"/> Mon. <input type="checkbox"/> Tues. <input type="checkbox"/> Wed. <input type="checkbox"/> Thurs. <input type="checkbox"/> Fri. <input type="checkbox"/> Sat.
------------------------	---	---	---------------------------	--

21. Date of Injury	Mo. Day Yr.	22. Date notice received	Mo. Day Yr.	23. Date stopped work	Mo. Day Yr.	<input type="checkbox"/> a.m. <input type="checkbox"/> p.m.
24. Date pay stopped	Mo. Day Yr.	25. Date 45 day period began	Mo. Day Yr.	26. Date returned to work	Mo. Day Yr.	<input type="checkbox"/> a.m. <input type="checkbox"/> p.m.

27. Was employee injured in performance of duty? ☐ Yes ☐ No (If "No," explain)

28. Was injury caused by employee's willful misconduct, intoxication, or intent to injure self or another? ☐ Yes (If "Yes," explain) ☐ No

29. Was injury caused by third party? <input type="checkbox"/> Yes <input type="checkbox"/> No (If "No," go to item 31.)	30. Name and address of third party (Include city, state, and zip code)

31. Name and address of physician first providing medical care (Include city, state, zip code)	32. First date medical care received	Mo. Day Yr.
	33. Do medical reports show employee is disabled for work?	<input type="checkbox"/> Yes <input type="checkbox"/> No

34. Does your knowledge of the facts about this injury agree with statements of the employee and/or witness? ☐ Yes ☐ No (If "No," explain)

35. If the employing agency controverts continuation of pay, state the reason in detail.	36. Pay rate when employee stopped work
	\$ Per

**Signature of Supervisor and Filing Instructions**

37. A supervisor who knowingly certifies to any false statement, misrepresentation, concealment of fact, etc., in respect of this claim may also be subject to appropriate felony criminal prosecution.

I certify that the information given above and that furnished by the employee on the reverse of this form is true to the best of my knowledge with the following exception:

Name of supervisor (Type or print)

Signature of supervisor Date

Supervisor's Title Office phone

38. Filing instructions

☐ No lost time and no medical expense: Place this form in employee's medical folder (SF-66-D)

☐ No lost time, medical expense incurred or expected: forward this form to OWCP

☐ Lost time covered by leave, LWOP, or COP: forward this form to OWCP

☐ First Aid Injury

**APPENDIX C3**

**TVA FORM 9179 (4/88)**

**CLAIMS OF DISABILITY FOR WORK DUE TO JOB-RELATED INJURY**

**CLAIMS OF DISABILITY FOR WORK DUE TO JOB-RELATED INJURY:  
NOTICE OF EMPLOYEE'S RESPONSIBILITIES**

According to regulations of the Federal Employees' Compensation Act (FECA), as revised effective June 1, 1987, if you are claiming disability for work due to a job-related injury, you have certain obligations as listed below.

1. **FILE A CLAIM PROMPTLY** - Complete the employee's side of claim form OWCP CA-1 and submit it to your supervisor as soon as possible, but **NO LATER THAN THIRTY DAYS AFTER THE DATE OF INJURY**.
2. **SUBMIT MEDICAL EVIDENCE** - Submit to TVA within 10 workdays medical evidence of disability for work due to the claimed injury.
3. **INFORM YOUR DOCTOR OF TEMPORARY LIGHT DUTY** - Inform your doctor of any offer by TVA to provide temporary light duty, where possible, to accommodate medical constraints imposed by the claimed injury.
4. **INFORM YOUR DOCTOR OF ALTERNATE JOBS** - Inform your doctor of any particular alternate jobs made available by TVA, and furnish the doctor with any written description of the specific duties and physical requirements of such jobs furnished by TVA.
5. **INFORM TVA IMMEDIATELY OF ANY MEDICAL LIMITATIONS OR CONSTRAINTS SPECIFIED BY YOUR DOCTOR**.
6. **RETURN TO WORK** - You are obligated to return to regular duty as soon as you are able to do so. **ALSO**, you are obligated to accept suitable offers by TVA of temporary light duty or alternate jobs not in conflict with medical limitations caused by the claimed injury.
7. **REPORT ALL EMPLOYMENT AND SELF EMPLOYMENT ACTIVITIES** - For all periods in which you claim COP or Compensation you are required to report all employment and self employment activities. You must report the activities performed and the income earned. Earned income for employment activities is defined as actual salary, wage, sales commissions, piecework commissions, and other payments of value such as housing allowances, meals, food, clothing, equipment, reimbursed expenses, etc. Additionally, if you performed activities in connection with a relative's or spouse's business, you must report as earned income what it would have cost the employer or organization to hire someone to perform the work you performed. For self employment activities, earned income is defined as the gross income received from the activity. If the self employment activity was operated at a loss or if the profits were reinvested you must report what it would cost to hire someone to perform the work you performed.

According to the FECA, WHERE AN EMPLOYEE REFUSES SUITABLE WORK offered by the employing agency according to FECA regulations, ENTITLEMENT TO COP CEASES as of the effective date of availability of such work.

Where an employee FAILS TO SUBMIT THE REQUIRED MEDICAL EVIDENCE WITHIN 10 WORKDAYS or REFUSES SUITABLE WORK, COP SHALL BE TERMINATED.

I have been informed of and understand the employee's responsibilities listed above.

\_\_\_\_\_  
EMPLOYEE'S SIGNATURE

\_\_\_\_\_  
DATE

\_\_\_\_\_  
OFFICIAL SUPERIOR'S SIGNATURE

\_\_\_\_\_  
DATE

PENALTIES UNDER 20 CFR, SECTION 10.23 ARE SHOWN ON THE BACK OF THIS SHEET.

## PENALTIES FOR FRAUD

UNDER THE FEDERAL EMPLOYEES' COMPENSATION ACT. PUBLISHED IN 20 CFR PART 10 Federal Register/Vol. 52, No. 62/Wednesday, April 1, 1987, p 10505

### Rules And Regulations. Section 10.23 Penalties

- (a) Any employee, beneficiary, official superior, representative, or other person who knowingly makes, or knowingly certifies to, any false statement, misrepresentation, concealment of fact, or any other act of fraud with respect to a claim under the Act, or who knowingly accepts compensation to which that person is not entitled, is subject to criminal prosecution and may, under appropriate U.S. Criminal Code provisions (e.g., 18 U.S.C. 287 and 1001), be punished by a fine of not more than \$10,000 or imprisonment for not more than five years, or both.
- (b) Any employee, beneficiary, official superior, representative, or other person who, with respect to a claim under the Act, enters into any agreement, combination, or conspiracy to defraud the United States by obtaining or aiding to obtain the payment or allowance of any false, fictitious and fraudulent claim is subject to criminal prosecution and may, under appropriate U.S. Criminal Code provisions (e.g., 18 U.S.C. 286), be punished by a fine of not more than \$10,000 or imprisonment for not more than ten years, or both.
- (c) Any person charged with the responsibility of making reports in connection with an injury who willfully fails, neglects, or refuses to do so; induces, compeis, or directs an injured employee to forego filing a claim; or willfully retains any notice, report, or paper required in connection with an injury, is subject to a fine of not more than \$500 or imprisonment for not more than one year, or both.

I have been informed of and understand the penalties printed above.

---

EMPLOYEE'S SIGNATURE

---

DATE

**APPENDIX C4**

**TVA FORM 255 (1/90)**

**REPORT OF VEHICLE ACCIDENT, THEFT, OR FIRE**

**(TVA VEHICLE ONLY)**

## REPORT OF VEHICLE ACCIDENT, THEFT, OR FIRE

**Instructions**

Prepare report immediately after occurrence. For accident or fire involving TV-00001 through TV-49999 send 3 copies to Transportation Services, Chattanooga and for TV-50000 through TV-99999 send 3 copies to Heavy Equipment Department, Chattanooga. For theft send 2 copies to Transportation Services or Heavy Equipment Department as appropriate and third copy to nearest TVA Public Safety Office. Always send 1 copy to employee's supervisor. Supervisor completes Supplement (pages 3 and 4) and distributes all four pages according to the instructions at the bottom of page 4

**TVA DRIVER**

Payroll Name	Age	Social Security Number	TVA Telephone
Plant, Facility, Department			Group or Organization
Supervisor's Name	Title	TVA Address	TVA Telephone
Responsible Manager	Title	TVA Address	TVA Telephone

**TIME AND LOCATION**

Date Occurred	Hour	Street or Road	City and State
---------------	------	----------------	----------------

**TVA VEHICLE**

Make	Model	Body Type	License Number
------	-------	-----------	----------------

**DAMAGE TO VEHICLE**

Description of Damage	
	Estimated Amount
	\$

**DISPOSITION OF TVA VEHICLE**

\_\_\_\_ Still in Service \_\_\_\_\_ Left at (TVA garage or other)

**PROPERTY OF OTHERS**

Kind Of Property And Extent Of Damage				
				Estimated Amount
				\$
If Automobile	Make And Year	Body Type	License Number	State
Owner Of Property		Address		Telephone
Where Property Can Be Seen				

**OTHER DRIVER**

Other Car Driver's Name	Address	Occupation	Telephone
Insurance Carried By		Kind Of Insurance	
____ Driver ____ Owner ____ None		____ Liability ____ Collision ____ Medical	
Name Of Insurance Company			

**PASSENGERS**

Full Name	Age	Address	TVA NONTVA
1			
2			
3			

**OTHER WITNESSES**

1	
2	
3	

**PERSONS INJURED IN ACCIDENT**

1	
2	
3	
4	
Nature And Extent Of Injuries	
Doctor's Name	Address
Place Injured Were Taken	

	Lights On (specify which)	Signals Given (specify which)	Speed (mph)		
			When Danger First Noticed	At Impact	Legal Limit
TVA VEHICLE (Owned or rented)					
OTHER VEHICLE					
TVA Restraining Devices	Lap Belt Installed Lap Belt Worn	Yes No <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	Shoulder Belt Installed Shoulder Belt Worn	Yes No <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	Prevented Injury <input type="checkbox"/> <input type="checkbox"/>
HIGHWAY CONDITIONS	Blacktop On grade	Concrete Level	Gravel-dirt Curve	Wet Open country	Dry Residential Ice-snow Commercial
LIGHT	Daylight	Night	Dusk-dawn	Streets lighted	
WEATHER	Clear	Rain	Snow	Fog	
TRAFFIC CITATIONS	OFFENSE CHARGED TO YOU	DISPOSITION	OFFENSE CHARGED TO OTHER DRIVER	DISPOSITION (If Known)	
INVESTIGATING OFFICER	NAME	State	City	County	TVA
SKETCH ACCIDENT ON DIAGRAM	<p>1. Write in street names or numbers. 2. Show traffic signs and control devices. 3. Show lanes, double yellow lines, center strips, etc. 4. Show direction and distance to nearest town, major intersection, and landmarks. 5. Draw and number vehicles involved and parked.</p> <p>6. Use solid line to show path before accident and broken line after accident.</p> <p>7. Show pedestrian.</p> <p>8. Show railroad.</p> <p>9. Show skid marks, and give lengths.</p> <p>10. Attach any photos.</p> <p>Indicate North by arrow in circle</p>				
DESCRIPTION OF ACCIDENT THEFT OR FIRE (Use additional sheets if necessary)					
PRIVACY ACT OF 1974	<p>In compliance with the Privacy Act of 1974, the following information is provided: Collection of the information is authorized by the Tennessee Valley Act of 1933, 16 U.S.C. 831dd and 40 U.S.C. 491. Disclosure of information is required by TVA regulations (TVA INSTRUCTION II TRANSPORTATION, Equipment). An employee of a federal agency who fails to report accurately a motor vehicle accident involving a federal vehicle may be subject to administrative sanctions. The principal purposes for collecting this information are: (1) To provide necessary data for use by legal counsel in any actions resulting from the accident, and (2) To provide accident information and statistics for use in analyzing accident causes and developing methods of reducing accidents. Routine uses include disclosure to federal, state, and local governments and agencies when relevant to civil, criminal, administrative, and regulatory investigations, actions, and proceedings.</p>				

SIGNATURE OF DRIVER

DATE

**SUPPLEMENT**  
**ADMINISTRATIVELY CONFIDENTIAL WHEN COMPLETED**  
**CONFIDENTIAL OPINION(S) AND RECOMMENDED ACTION(S) TO**  
**PREVENT RECURRENCE OF**  
**VEHICLE ACCIDENT, THEFT, OR FIRE**

**SUPERVISOR'S REVIEW, OPINIONS, AND RECOMMENDATION(S)**

Supervisor completes this supplement and distributes according to instructions on page four.

Reviewer's Name \_\_\_\_\_ Date \_\_\_\_\_ Date of Accident \_\_\_\_\_

What was happening before the accident?

☐ 01 Roadway driving☐ 03 Vehicle being serviced☐ 02 Off-the-road driving☐ 04 Vehicle idle

What type of vehicle?

☐ 001 Passenger car/van/wagon☐ 002 Pickup truck☐ 003 Compact car \_\_\_\_\_☐ 004 Compact pickup truck☐ 005 Tractor-truck☐ 006 Other truck☐ 007 Other type vehicle (specify) \_\_\_\_\_

(Accidents involving damage to other types of vehicles such as industrial forklifts, mowers, scooters, construction equipment not being used as a motor vehicle at the time of the accident, etc., should be reported on form TVA 18002, Report of Accidental Property Damage, Fire, or Fire Related Incident)

An accident is "Driver-Controllable" if in your opinion the TVA driver could likely have prevented the accident through prudent actions. In your opinion was this accident "Driver-Controllable"? ☐ Yes ☐ No

Why? \_\_\_\_\_

What immediate actions do you recommend be taken to prevent recurrence of a similar accident?

	Recommended Action	Person Responsible	Completion Date
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____

What long-term action(s) do you recommend be taken to prevent a recurrence of a similar accident?

	Recommended Action	Person Responsible	Completion Date
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____

Reviewer signature

Next higher level management signature  
and approval of recommended action(s)

Date \_\_\_\_\_

Date \_\_\_\_\_



Use for continuation of narrative or other information which in your opinion is of importance.

**Distribution** (Send copies of all four pages to):

OC H&S. MPB 1E 207B-M

TVA General Counsel, Knoxville

Transportation Services, Chattanooga (TV-00001 - TV-49999)

Appropriate Heavy Equipment Department (TV-50000 - TV-99999)

Appropriate Responsible Manager

**APPENDIX C5**

**FORM SR-13 (1/93)**

**ALABAMA DEPARTMENT OF PUBLIC SAFETY**

**ACCIDENT INVOLVING A PRIVATE VEHICLE**

## COMPLETION OF THIS FORM IS REQUIRED BY §32-7-1, CODE OF ALABAMA 1975.

FAILURE TO FILE A REPORTABLE ACCIDENT ON THIS FORM MAY RESULT IN SUSPENSION OF YOUR DRIVER LICENSE.

**INFORMATION AND INSTRUCTIONS:** Completion of this form is required ONLY if a motor vehicle accident occurring in Alabama caused death, personal injury, or property damage to any one owner in excess of \$250. The driver of any motor vehicle, which is in ANY MANNER involved in an accident in this state, is legally required to file a report on this form with the Department of Public Safety within ten (10) days after the accident regardless of whether or not at fault and regardless of whether or not the vehicle involved was covered by liability insurance at the time of the accident. If such driver is physically incapable of making such report, the owner of the motor vehicle involved in such accident shall, within ten (10) days after learning of the accident, make such report.

DATE OF ACCIDENT	<input type="checkbox"/> A.M. <input type="checkbox"/> P.M.	NO. OF VEHICLES	For Office Use Only		
LOCATION OF ACCIDENT (ST./HIGHWAY)		COUNTY	Subject	Injuries	Claims

## VEHICLES INVOLVED

YOUR INFORMATION (PLEASE PRINT OR TYPE)				OTHER PARTY'S INFORMATION (PLEASE PRINT OR TYPE)					
YOU ARE THE: <input type="checkbox"/> DRIVER <input type="checkbox"/> PEDESTRIAN <input type="checkbox"/> PROPERTY OWNER <input type="checkbox"/> OTHER				OTHER PARTY WAS: <input type="checkbox"/> DRIVER <input type="checkbox"/> PEDESTRIAN <input type="checkbox"/> PROPERTY OWNER <input type="checkbox"/> OTHER					
NAME (FIRST, MIDDLE, LAST)		TELEPHONE NO.		NAME (FIRST, MIDDLE, LAST)		TELEPHONE NO.			
ADDRESS: STREET NO.				ADDRESS: STREET NO.					
CITY		STATE	ZIP	CITY		STATE	ZIP		
DRIVER'S DATE OF BIRTH	SEX <input type="checkbox"/> M <input type="checkbox"/> F	DRIVER LICENSE NO.	STATE	DRIVER'S DATE OF BIRTH	SEX <input type="checkbox"/> M <input type="checkbox"/> F	DRIVER LICENSE NO.	STATE		
NAME OF OWNER		IF SAME AS DRIVER, MARK BOX <input type="checkbox"/>		NAME OF OWNER		IF SAME AS DRIVER, MARK BOX <input type="checkbox"/>			
ADDRESS OF OWNER: STREET NO.				ADDRESS OF OWNER: STREET NO.					
CITY		STATE	ZIP	CITY		STATE	ZIP		
OWNER'S BIRTH DATE	SEX <input type="checkbox"/> M <input type="checkbox"/> F	OWNER'S DRIVER LICENSE NO.	STATE	OWNER'S BIRTH DATE	SEX <input type="checkbox"/> M <input type="checkbox"/> F	OWNER'S DRIVER LICENSE NO.	STATE		
YOUR VEHICLE				OTHER VEHICLE (Use additional form if more than two [2] vehicles)					
YEAR	MAKE	TYPE	COMMERCIAL <input type="checkbox"/> YES VEHICLE <input type="checkbox"/> NO	STATE	YEAR	MAKE	MODEL	TYPE	STATE
VIN		LICENSE PLATE NO.			VIN		LICENSE PLATE NO.		

## PROPERTY DAMAGE

DESCRIPTION OF PROPERTY DAMAGE (OTHER THAN VEHICLE)

## INJURED PERSONS (CLAIM FOR PERSONAL INJURY ON REVERSE)

FULL NAME OF INJURED IN YOUR VEHICLE		DID INJURED <input type="checkbox"/> YES DIE? <input type="checkbox"/> NO	
ADDRESS: STREET NO.			
CITY		STATE	ZIP
DATE OF BIRTH	SEX <input type="checkbox"/> M <input type="checkbox"/> F	INJURED WAS: (Please circle) 1 DRIVER 2 PASSENGER 3 PEDESTRIAN 4 OTHER	
FULL NAME OF INJURED IN YOUR VEHICLE		DID INJURED <input type="checkbox"/> YES DIE? <input type="checkbox"/> NO	
ADDRESS: STREET NO.			
CITY		STATE	ZIP
DATE OF BIRTH	SEX <input type="checkbox"/> M <input type="checkbox"/> F	INJURED WAS: (Please circle) 1 DRIVER 2 PASSENGER 3 PEDESTRIAN 4 OTHER	

## INSURANCE AND/OR SECURITY

Complete the following as required by the Safety Responsibility Law of Alabama (§32-7-1 and following sections). Mark only the appropriate box. All information will be verified.

- ☐ 1. No liability insurance in effect at time of accident.  
☐ 2. Form SR-23 (fleet policy) on file with DPS.  
☐ 3. Your vehicle is a qualified carrier with APSC. ☐ YES ☐ NO  
APSC Certificate No. \_\_\_\_\_  
☐ 4. Department of Public Safety Self-Insurance  
Certificate No. \_\_\_\_\_  
☐ 5. Motor vehicle liability policy issued by \_\_\_\_\_

(Name of insurance company, not agency)

POLICY NO. \_\_\_\_\_

POLICY PERIOD FROM \_\_\_\_\_ TO \_\_\_\_\_

POLICY HOLDER \_\_\_\_\_

SIGNATURE \_\_\_\_\_ DATE \_\_\_\_\_

(Complete Reverse Side)

**INFORMATION AND INSTRUCTIONS:** Complete this portion of the form if you believe that another party is responsible for your damages. Do not delay filing this form because amount of damages is unknown.

**PROPERTY DAMAGE**

I, \_\_\_\_\_ [Full Name of Person Making Claim] \_\_\_\_\_  
certify that damages to my property amounted to \$ \_\_\_\_\_ [Amount of Damage]  
as a result of this motor vehicle accident. I believe I am entitled to recover the amount specified above from  
\_\_\_\_\_ [Driver of Vehicle] and from  
\_\_\_\_\_ [Owner(s) of Vehicle], and I have not released said party(ies).  
Signature of Property Owner \_\_\_\_\_ Date \_\_\_\_\_

**INJURIES (Please complete one section for each party injured.)**

I, \_\_\_\_\_ [Full Name of Person Injured] \_\_\_\_\_  
certify that my medical expenses are \$ \_\_\_\_\_ [Amount of Damage]  
as a result of this motor vehicle accident. I believe I am entitled to recover the amount specified above from  
\_\_\_\_\_ [Driver of Vehicle] and from  
\_\_\_\_\_ [Owner(s) of Vehicle], and I have not released said party(ies).  
Signature of Claimant /Legal Guardian of Minor \_\_\_\_\_ Date \_\_\_\_\_

I, \_\_\_\_\_ [Full Name of Person Injured] \_\_\_\_\_  
certify that my medical expenses are \$ \_\_\_\_\_ [Amount of Damage]  
as a result of this motor vehicle accident. I believe I am entitled to recover the amount specified above from  
\_\_\_\_\_ [Driver of Vehicle] and from  
\_\_\_\_\_ [Owner(s) of Vehicle], and I have not released said party(ies).  
Signature of Claimant /Legal Guardian of Minor \_\_\_\_\_ Date \_\_\_\_\_

I, \_\_\_\_\_ [Full Name of Person Injured] \_\_\_\_\_  
certify that my medical expenses are \$ \_\_\_\_\_ [Amount of Damage]  
as a result of this motor vehicle accident. I believe I am entitled to recover the amount specified above from  
\_\_\_\_\_ [Driver of Vehicle] and from  
\_\_\_\_\_ [Owner(s) of Vehicle], and I have not released said party(ies).  
Signature of Claimant /Legal Guardian of Minor \_\_\_\_\_ Date \_\_\_\_\_

**FORM COMPLETION REVIEW**

1. Review form to ensure all blanks have been filled in.
2. Use your full, legal name.
3. Describe all property damage (Ex.: bicycle, farm equipment, house, fence, etc.).
4. Sign and date this form in spaces provided.

**APPENDIX C6**

**FIRST AID INSTRUCTIONS FOR TVA EMPLOYEES**

# **FIRST-AID INSTRUCTIONS**

**For  
TVA Employees**

**TENNESSEE VALLEY AUTHORITY**

**Division of Medical Services**

**1986**

**TVA/OCS/MS-86/1**

## ***Introduction***

The instructions in this booklet have been developed to assist TVA employees in using first-aid measures safely and confidently, and to furnish them with specific information regarding the use of supplies available in TVA first-aid kits and first-aid rooms.

This material is not intended as a first-aid text, nor is it a substitute for first-aid training. It is a composite of simple procedures for handling some of the more common injuries and ailments encountered both on and off the job. Employees are encouraged to participate in first-aid training courses offered by TVA and to hold up-to-date first-aid certificates.

All first-aid treatment of injuries on the job, regardless of severity, must be reported according to the procedure described in the TVA Instruction under VIII INJURY, Employee.

First-aid kits, supplies, and refills for locations where there are medical offices or health stations (*except* Chattanooga) may be obtained on TVA form 9275 submitted to the local medical office or health station. At other locations, and at Chattanooga, storeroom requisitions (forms 9275) should be sent to the Medical Director.

Supplies for refills for first-aid kits also are available at project medical offices, steam plant health stations, and certain district and division offices of the Office of Power.

REMEMBER THAT THE OBJECT OF FIRST AID IS TO MAKE THE PERSON MORE COMFORTABLE AND TO REDUCE THE CHANCE OF FURTHER INJURY OR DISABILITY. WHEN IN DOUBT ALWAYS REFER TO A PHYSICIAN.

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## ABRASIONS AND SCRATCHES

- Wash gently but thoroughly with soap and water.
- Rinse with clean water.
- Remove obvious foreign particles from the wound.
- Apply antiseptic.
- Apply sterile dressing.

## ACUTE ILLNESS

Headache, cough, chills, dizziness, nausea, vomiting, sore throat, or fever may be forerunners of a communicable or severe disease. They may also accompany exposures to various poisonous materials. If these symptoms are severe enough to cause complaint, medical advice should be sought. Other workers should be protected from exposure by isolating the sick worker until he can be seen by a physician.

## AMPUTATION

- In case of partial or complete amputation every effort should be made to preserve the severed part. Hold it in position with a sterile compress and support with splint.
- Control bleeding as quickly as possible.
- Use a sterile compress to help control bleeding.
- Use a tourniquet only if direct pressure and all other procedures to control bleeding fail.
- Place a sterile dressing over all injured tissue.
- Treat injured person for shock (See p. 13.)
- Refer to physician immediately.

## ANIMAL BITES (INCLUDES BAT BITES)

- Wash wound with soap and water to remove animal saliva.
- Rinse well.
- Apply sterile dressing.
- Refer to physician.
- Notify local health department.

### Caution:

*It is not necessary to kill domestic pets suspected of having rabies, except to protect others from danger. Such pets should be confined and observed for 10 days for such signs of rabies as changed behavior, excitability, salivation, paralysis, and death. Wild animals may not show clear signs of rabies and*  
(Continued)

*should be killed for examination.*

*If you have to kill a suspected animal, try not to damage its head.*

*If you are bitten by an animal, always suspect it to be rabid until proved otherwise.*

## ASPHYXIA (STOPPAGE OF BREATHING)

### *Common Cause:*

Obstruction of air passage to lungs as in choking, drowning, etc.

Paralysis of the respiratory center of brain as in electric shock.

Interference with the oxygen-carrying function of red blood corpuscles as in carbon monoxide asphyxiation.

Lack of oxygen in air breathed as in storage bins, mines, etc.

### *Treatment:*

Be sure person is in fresh air.

Start artificial respiration immediately. Follow instructions on pages 17-18.

Treat for shock. (See p. 13.)

Call for help at once.

## BRUISES

A bruise is usually caused by a blow or fall. The skin is not broken but the tissues underneath are injured, resulting in broken small blood vessels. Pain, swelling, and black and blue colors appear.

### *Treatment:*

Immediately apply cloths wrung out in cold water or an icebag. If possible, elevate the injured part and place it at complete rest.

The general rule is "cold" applications for the first 24 hours followed by "heat."

If soreness or disability persists, refer patient to physician.

## BURNS AND SCALDS

### FIRST and SECOND DEGREE THERMAL BURN

Immerse burned area in a container of cold water, preferably 45-50° F. If the part cannot be immersed, apply cloths soaked in ice water and change the cold packs constantly.

Continue cold-water treatment until patient can stand removal without recurrence of pain.

Do not break blisters intentionally.

Treat the person for shock (see page 13.) (Continued)

After cold-water treatment, cover burn area with sterile dressing.

Refer patient to physician.

### THIRD DEGREE THERMAL BURN

Cover with dry sterile dressing.

Treat for shock (see page 13.)

Leave blisters alone.

Refer to physician.

### CHEMICAL BURNS: (Acid or Alkali)

Flush burned area at least 15 minutes with large quantities of water (preferably lukewarm).

Cover with sterile dressing.

Refer patient to physician if burns are severe or extensive.

### CREOSOTE BURNS:

Use creosote burn wash found in TVA first-aid kits.

### ELECTRIC BURNS:

Should always be treated as severe burns, unless clearly of a minor nature.

EYE BURNS: (See page 7.)

### SUNBURN:

Same treatment as for minor burn, if small area involved.

Severe or extensive cases should be referred to a physician.

## CARBON MONOXIDE POISONING

Remove victim to fresh air.

If breathing has stopped or comes in gasps, start artificial respiration (see page 17) and continue until natural breathing is restored or until the doctor pronounces person dead.

Keep victim warm and insist on complete rest until he is seen by a physician. Even slight exercise is dangerous.

Refer to a physician.

## CHEST AND ABDOMINAL INJURIES

Blows to the chest and abdomen may result in injury to underlying organs and tissues, even though no sign of injury may be seen.

(Continued)

*Treatment:*

- Keep patient warm and quiet.
- Do not move until transportation is arranged.
- Cover open wounds with sterile dressings.
- In case of suspected abdominal injury give nothing by mouth.
- Refer to a physician.

**CHOKING**

If the victim is breathing well with only partial obstruction and is still able to speak or cough effectively, do not interfere with his attempts to expel a foreign body. If the victim cannot speak or cough, uses a distress signal, appears blue, or shows an exaggerated effort to breathe, you must follow the procedures described below.

**SITTING OR STANDING VICTIM**

Stand behind the victim and wrap your arms around his waist. Place the thumb side of your fist against the victim's abdomen, slightly above the navel and below the xiphoid process (tip of the breastbone). Grasp your fist with your other hand and press it into the victim's abdomen with four quick upward thrusts.

**SUPINE VICTIM**

Place one of your hands on top of the other, with the heel of the bottom hand in the middle of the victim's abdomen, slightly above the navel and below the rib cage. Move forward so that your shoulders are directly over the victim's abdomen and press upward toward the diaphragm with four quick thrusts. Do not press to either side.

**COLD INJURY****CHILBLAIN:***Prevention:*

- Keep feet warm and dry.
- Avoid standing for a long time without exercise.

*Treatment:*

- Immerse in warm, not hot, water.
- Treat as a minor burn.

(Continued)

**"TRENCH" FOOT:**

After hours of exposure to low (but not freezing) temperature, feet become very painful. Swelling and numbness follow.

*Treatment:*

- Keep patient off his feet. Use a stretcher to carry him to a physician.
- Cover foot (or feet) with sterile dressing.
- Do not allow victim to smoke.

**FROST BITE:**

Signs of frostbite are whiteness and numbness of the flesh; the skin feels cold to the touch.

*Treatment:*

- For small areas such as the nose, ears, and other parts of the face, place warm palm of hand over area, but do not rub.
- Frostbitten fingers: Warm directly against skin in armpit.
- Frostbitten feet: Immerse in warm (but NOT hot) water.
- Encourage gentle exercise of fingers and toes.
- Don't expose to high temperature immediately.
- Give victim a warm drink.
- Handle frozen part with great care to avoid injury to it.
- Refer to physician if injury is severe or extensive.

**COMMUNICABLE DISEASES**

Communicable diseases are most catching in the early stages, even before rash and other signs appear. Therefore, if a worker knows he has been exposed to a communicable disease, he should take personal responsibility not to spread it. He should consult a physician immediately upon the onset of such warning signs as cough, sore throat, aching in joints and muscles, unusual tiredness, and the like.

**CONVULSIVE SEIZURE (EPILEPSY)**

Insert, but do not force, a small piece of wood well wrapped with gauze or clean cloth between patient's teeth to keep him from biting his tongue.

- Do not attempt to hold patient still. Protect him from injury as he thrashes about.
- Give nothing by mouth.
- Refer to physician.

## DERMATITIS (SKIN RASH)

Some skin diseases may be caused by things individuals come in contact with either on the job or elsewhere. Preventing contact with materials to which one is sensitive will help control skin rashes.

### *Prevention:*

- Use engineering safeguards and protective clothing which have been recommended.
- Practice good housekeeping in your work area.
- Keep clean—personal cleanliness is the most important single factor in control of contact dermatitis.
- Dry hands well after washing.
- Do not use kerosene, gasoline, or carbon tetrachloride or other similar solvents for cleansing skin.
- Ointments and lotions may aggravate dermatitis unless such treatment is prescribed for specific conditions.
- Remove contaminated clothing as soon as possible.

### *Treatment—*

- Refer to physician.

## DIABETIC EMERGENCIES

Unconsciousness is sometimes a complication in the person with diabetes. This may be of two types—one due to the disease and the other due to temporary accumulation in the body of too much insulin which has been used in the control of the disease.

In either case, the unconscious diabetic should be seen by a physician without delay.

Every diabetic should carry an identification card and should inform at least one of his fellow employees of his disease. Diabetes often complicates the healing of wounds. Injuries to the feet are especially troublesome and should be treated by a physician. All other injuries should be treated by a physician if healing does not take place promptly.

## DISLOCATIONS

The injured joint looks out of shape when compared with a similar joint, and there is pain and usually swelling. Many dislocations are also accompanied by broken bones. (Continued)

### *Treatment:*

- Do not attempt to put a dislocation back in place.
- Support dislocations in a comfortable manner.
- Treat for shock if necessary (See page 13.)
- Refer to physician.

## DROWNING

Make certain that air passages are not blocked.

Start artificial respiration at once, check for circulation, and continue resuscitation as needed until physician declares there is no further need, or until you become exhausted. (See pages 17 & 18.)

Treat for shock.

Send for physician.

When breathing is restored make sure he receives medical attention by a physician.

## EYES

CHEMICAL BURNS: (Acid or Alkali)

### *Treatment:*

Wash the eye freely with clean water. This may be done by immersing the face in pan or bowl of water, gently pulling back the lids, and moving the eye back and forth. Another way is to place the person on his back, hold his eyelids open with your fingers, and pour water into the inner corner of the eye from a pitcher or other container.

Continue this for at least 15 minutes.

Apply eye patch.

Refer to physician as soon as possible.

### *Caution:*

When work is being done in which there is a possibility of chemical burn, be sure there is plenty of clean water available.

### FOREIGN BODY:

*On Surface of the Eyeball:*

Do not try to remove.

Refer to physician.

*On Surface of the Membrane Lining the Eyelid:*

Pull the lower lid down gently and look for the speck.

(Continued)

Remove it with a corner of clean piece of gauze or a twist of cotton moistened with water.

If speck seems to be on the lining of the upper eyelid, grasp the lashes of the upper lid gently and draw the upper lid down over the lower lid. As the upper lid returns to its normal position, the foreign body may be caught on the lashes of the lower lid and removed by washing action.

If these measures fail, refer to a physician.

#### EYE INJURIES:

If the eye is injured by a foreign body—like a splinter of glass, metal, or wood, or by a particle blown into it with great force—apply eye patch.

If there is a protruding foreign body, do not remove it but bandage both eyes, using great caution so that the foreign body is not driven into the eyeball.

If the eyelids and tissue around them are injured, apply a firm bandage to prevent movement of the lids.

Refer to a physician.

### FAINTING

#### *Prevention:*

If a person says he feels as if he will faint, have him sit down and bend his body forward until his head is level with his knees. He should hold this position for a few minutes or lie down.

#### *Treatment:*

Place person on back with head lower than body.

Supply cool air.

Loosen clothing around neck and waist.

After consciousness returns, person should continue to lie quiet for a while before getting up.

If the faint lasts more than a few minutes the person should be referred to a physician.

### FRACTURE (BROKEN BONES)

#### ACTUAL OR SUSPECTED FRACTURE:

Gentleness is more important than speed in handling the patient.

Keep injured part at rest by splinting and bandaging.

Keep person warm and quiet.

Refer to physician.

#### COMPOUND FRACTURE:

If the bone shows through the skin, cover injured part with a sterile dressing. Control bleeding if present.

Do not disturb position of injured part. Splint.

Treat for shock (See page 13.)

Refer to physician.

### HEAD INJURY

#### *Treatment:*

Keep the person lying down.

Do not move the victim's head unless airway maintenance is necessary.

Treat any bleeding wound. (Bleeding from ears or nose may indicate serious injury. Do not attempt to treat.)

Keep patient warm and quiet.

Give nothing by mouth.

Refer to physician.

#### *Caution:*

If blow on head is hard enough to cause even momentary unconsciousness, refer to physician.

### HEAT CRAMPS

Same treatment and preventive measures as for heat exhaustion (see below).

### HEAT EXHAUSTION

This condition may occur during long heat waves or in locations where heavy work is done in high temperatures. The victim may show signs of shock and usually is conscious, pale, and cool.

#### *Prevention:*

Drink water often throughout the day, 1 glass at a time.

Wear working clothes that are light and porous to promote evaporation of perspiration. (Continued)

**Treatment:**

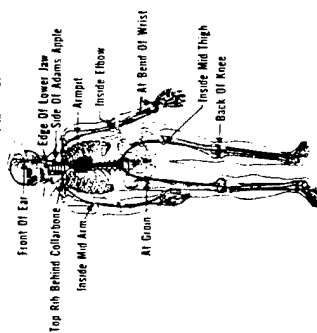
- Have person lie down in cool place with head lowered.
- Loosen his clothing.
- Cover body with light covering.
- If he is conscious give cool water.
- Refer to physician if symptoms do not disappear quickly.

**HEAT STROKE OR SUN STROKE**

Heat stroke is characterized by a flushed face and hot skin. Person often becomes unconscious. (See page 16.)

**Treatment:**

- Get person to a cool place. Elevate the upper part of the body.
- Remove as much clothing as is necessary to apply cold cloths to head and body.
- Continue applying cold cloths until consciousness returns or until body temperature returns to near normal.
- Watch for signs of shock and treat if necessary. (See page 13.)
- When person is conscious, give cool water.
- Give NO stimulants.
- Refer to a physician as quickly as possible.

**HEMORRHAGE (External Bleeding)****PRESSURE POINTS TO CONTROL ARTERIAL BLEEDING**

Apply finger pressure on artery against underlying bone at a pressure point immediately above wound.

- Use tourniquet ONLY when other control methods for bleeding have failed.

Refer to physician.

(Continued)

**TOURNIQUET WARNING:**

- Once the tourniquet has been applied leave it until a physician removes it.
- Make certain tourniquet is tight enough to stop bleeding.
- Do not apply dressing over tourniquet.
- A notation should be made and attached to the injured person giving the site of the tourniquet and time of application.

**INSECT BITES**

- To relieve the discomfort caused by the bite or sting of bees, mosquitos, wasps, or flies, apply a Sting-Kill swab if a first-aid kit is available.
- Alternate procedure:* Apply weak ammonia water or a paste of baking soda and water.
- If the stinger is left in the wound, withdraw it.
- If swelling and pain persist, refer to a physician.
- Any individual with a history of being sensitive to insect bites should be sent to a physician immediately.*

**NOSEBLEED**

- Have person sit quietly with head tilted slightly forward to prevent blood from accumulating in throat.
- Loosen collar.
- Wring out cloths wet with cold water and apply over nose, pressing the nostril on the bleeding side against the central portion of the nose for 4 or 5 minutes.
- Instruct person not to blow nose for hour or two after bleeding has stopped.
- Severe or repeated nosebleed requires medical attention.

**POISONS**

- Identify the poison, estimate the amount taken, and save all containers to assist treatment. After initial emergency care has been given, get the victim to a hospital without delay.
- Administer artificial respiration, if needed.
- Keep victim warm and quiet.
- Take other actions advised by Poison Control Center.

(Continued)

**DO NOT** induce vomiting *IF*:

- a) The victim has ingested strong corrosives which have burned the mouth and throat, or has ingested petroleum products (kerosene, gasoline);
- b) The victim is unconscious, semi-conscious, convulsing or has convulsed;
- c) The victim is pregnant; or
- d) The victim has severe heart disease.

In most other cases, prompt cleansing of the stomach through vomiting is indicated. Collect and save vomitus for hospital evaluation.

To induce vomiting, administer 1 tablespoon of *syrup* of ipecac followed by several glasses of warm water or soft drinks. (Vomiting is more effective if stomach is partially full.)

Vomiting usually begins in five to fifteen minutes. If syrup of ipecac is not available, vomiting may be induced by tickling the back of the victim's throat with a finger or the blunt end of a spoon, fork or knife.

Keep victim's head low and turned to side during vomiting to prevent his breathing in vomitus.

If victim is conscious but lips, mouth, and tongue ARE **STAINED AND BURNED** by corrosives, acid or alkali:

Do NOT force vomiting.

Dilute by having the victim drink a glass of water or milk if he is conscious and not having convulsions. Discontinue dilution if it makes him nauseated.

Refer to a hospital or physician without delay.

**POISONING FROM CONTAMINATED FOOD:***Prevention:*

Keep lunch as cool as possible, out of sun and away from any heat source.

Be careful what you carry in lunch, particularly in hot weather.

Medical office personnel can advise you about this.

*Treatment:*

Give lukewarm water by mouth to help flush out stomach.

Treat for shock. (See page 13.)

Refer to a physician.

**POISON IVY, POISON OAK, OR POISON SUMAC***Prevention:*

Be able to recognize plant and avoid contact with it.

Wear long sleeves and trousers when there is a possibility of being exposed to the plant.

After exposure, you may be able to prevent rash by washing the exposed skin with laundry soap and water and then applying rubbing alcohol.

*Treatment:*

Wash thoroughly with soap and water.

Apply calamine ointment or lotion or caladryl to itching area.

If the eruption persists, spreads, or is severe, refer to a physician.

**PUNCTURE WOUNDS****SLIGHT:**

Encourage bleeding by mild pressure.

Wash with soap and water.

Apply sterile dressing.

Check to see whether person has had tetanus immunization.

**SEVERE:** (Wound that penetrates into underlying tissues)

Control bleeding with sterile compress.

Apply sterile dressing.

Refer to physician for treatment. This type of injury can result in tetanus and other serious infection.

**NOTE:** Tetanus (lockjaw) immunization is available at all TVA medical offices and health stations. All employees should receive this protection and should carry immunization records with them to enable doctors treating injuries to know whether to give anti-toxin or "booster" shots of toxoid. Cooperating examining physicians, when requested on form TVA 424, will also give booster shots.

**SHOCK****FOLLOWING INJURY:**

Control bleeding if present. (See page 10.)

Keep person comfortably warm.

(Continued)

Remove all foreign bodies from mouth.

Loosen tight clothing.

Place person in lying-down position with his feet higher than his head (except in chest injury or suspected head fracture).

Relieve pain as much as possible.

Give artificial respiration if indicated. (See page 17.)

Refer to a physician.

#### **ELECTRICAL SHOCK:**

Break contact with electrical conductor, turn off switch if possible. If this cannot be done, stand on a folded dry coat or newspaper or a dry board. With one hand protected by several thicknesses of dry cloth or newspaper, or with a dry stick or pole, grasp a dry part of victim's clothing and drag him away from the conductor. It may be possible to push a live wire off the victim with a dry wooden stick, or to pull the victim off a live wire with a piece of dry rope or your belt looped over the foot or hand.

If victim is not breathing, start artificial respiration immediately and check for circulation. Continue resuscitation as needed until breathing is restored or until physician indicates no further need or until you become exhausted. (See pages 17-18.)

Call for physician.

After victim is revived, apply sterile dressing to burns.

Refer to a physician.

### **SLIVERS AND SPLINTERS**

If the sliver is near the surface and can be grasped with the forceps or fingers, remove and treat the wound as a puncture wound. (See page 13.)

If the skin is deeply punctured by a foreign object, tetanus may result. (See page 13.)

Do not attempt to remove deeply imbedded objects.

Refer to a physician.

### **SNAKE BITE (Poisonous)**

Treat as follows:

1. The victim should remain calm, avoid exertion, and do nothing which would stimulate blood circulation (no alcohol).

(Continued)

2. Apply a constriction band two or three inches above the bite (between the bite and the heart) just tight enough to insert a finger beneath it. This band should not stop blood flow. This band should be left in place until medical help is obtained.
3. Wash the area around the fang marks with soap and water and gently squeeze any blood or venom from the wound that might be present.
4. Do not cut the skin or apply ice.

Snake antivenom is one of the most effective treatments for snakebites when administered by qualified professional personnel. However, there is considerable risk of severe allergic reactions to the drug and its use by lay personnel is not recommended.

### **SPRAINS**

Occur when ligaments supporting a joint or connecting bones are torn.

#### *Treatment:*

Elevate the injured part and apply cloths wrung out in cold water or an ice pack.

A firm cravat bandage made from a triangular bandage is useful in supporting the injured part until it can be examined by a physician.

If in any doubt as to the extent of the injury, treat it as a fracture.

Refer to a physician.

### **STRAINS**

Occur when muscle fibers are torn.

#### *Prevention:*

Proper lifting and use of body mechanics.

Get assistance in moving heavy weights.

#### *Treatment:*

Put injured part at rest.

Apply cloths wrung out in hot water.

Refer to a physician.



## TOOTH AND MOUTH INJURIES

### FRACTURED JAW:

- Gently place the jaw in position so that the upper and lower teeth are together.
- Stabilize jaw by supporting with a triangular bandage tied at the top of the head.
- Remove gauze if patient has any breathing difficulty or becomes nauseated or unconscious.
- Apply ice pack to affected side to control swelling and pain.
- Give available medication for severe pain.
- Refer to physician or hospital.

### FRACTURED AND INJURED TEETH:

- Isolate fractured or loose teeth with gauze pads.
- Close teeth to hold in place.
- Give available medication for pain.
- Refer to dentist.

### INJURIES TO TISSUES INSIDE MOUTH:

- Use standard methods for controlling bleeding as indicated by nature of wound. Wrap index finger in sterile gauze, apply direct pressure. Use cold packs for bruised lips.

#### Bleeding from recent extraction site:

- Place several layers of 2" gauze over bleeding site.
- Close teeth firmly together to apply pressure for 10-15 minutes; repeat as necessary.
- Refer to dentist.

### TOOTHACHE:

- Give available medication for pain.
- Do not place aspirin or other medication that is designed for ingestion on the tooth or gum.
- Refer to physician.

## UNCONSCIOUSNESS

Unconsciousness may be caused by a number of things such as heart failure, stroke, diabetic coma or insulin shock, epilepsy, excessive drinking, inhalation of toxic gases, head injuries, internal hemorrhage, etc.

Any unconscious person, except a person who has a head or neck injury, should have his neck lifted and his head tilted backward (Continued)

as in figure 2, page 17. If unconsciousness is due to a head injury the airway can be opened by thrusting the lower jaw forward into a jutting out position, the jaw thrust method. (Figure A, page 17).

If possible, determine the cause of unconsciousness and treat accordingly.

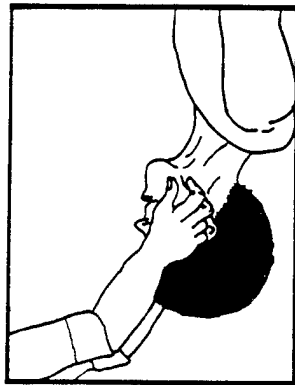
Give nothing by mouth.

Do not move the person more than is necessary.

If breathing has stopped, start artificial respiration.

Treat for shock. (See page 13.)

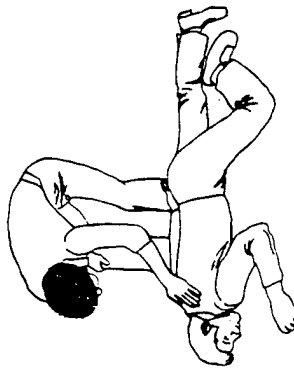
Get medical care at once.



(Figure A)

## CARDIOPULMONARY RESUSCITATION

1. **DETERMINE IF VICTIM IS UNCONSCIOUS.** Tap or gently shake victim's shoulder and shout - "Are you O.K.?" Call out - "Help!"



(Figure 1)

2. **OPEN AIRWAY.** Place one hand beneath the victim's neck and the other hand on the forehead. Gently lift the neck while pressing firmly on the forehead. This should open the airway. Place your ear near the victim's mouth and nose. **LOOK** at the chest for movement, **LISTEN** for breaths and **FEEL** for breathing against your cheek. If the victim is not breathing you should proceed to the next step.



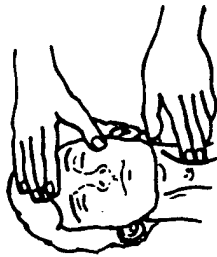
(Figure 2)

3. **GIVE FOUR QUICK FULL BREATHS.** Keep the head tilted and pinch the nose. (Continued)



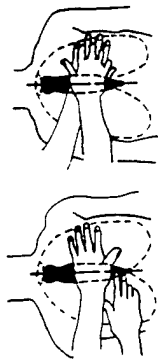
(Figure 3)

4. **CHECK FOR PULSE** - While keeping the head tilted with pressure on the forehead check the pulse for at least 5 seconds but no more than 10 seconds. Place your finger tips on the Adam's apple and slide your fingers into the groove at the side of the neck nearest you. If there is a pulse but no breathing give one breath every 5 seconds. If there is no pulse or breathing send someone to call an ambulance.



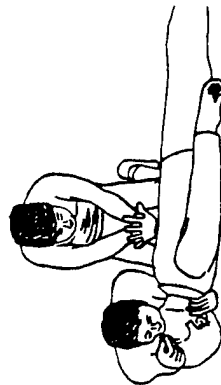
(Figure 4)

5. **FIND THE CORRECT HAND POSITION FOR CHEST COMPRESSIONS** - With your middle and index fingers trace the rib cage to the notch where the ribs and breastbone meet. Place the middle finger on the notch and the index finger next to it. Put the heel of other hand on the breastbone next to your fingers. Put the first hand on the top of the hand on the breastbone. Keep your fingers off the chest.



(Figure 5)

6. **PUSH 15 - BREATHE 2** - Give 15 compressions at a rate of 80 per minute. Tilt the head and give 2 quick full breaths. Continue to repeat 15 compressions followed by 2 compressions. Check the pulse and breathing after 1 minute and every few minutes thereafter.



(Figure 6)

## FIRST-AID KITS AND SUPPLIES

### FIRST-AID KITS

TVA  
Stock No.

First-Aid Kit, 16-unit size (filled) ..... 11375  
First-Aid Case for 24-unit kit (empty) ..... 11370  
(Order contents extra. This kit is for use only on heavy-duty line maintenance trucks.)

### SUPPLIES FOR FIRST-AID KIT

Item	TVA Stock Number	Number of Packages to 16-Unit Size	Number of Packages to 24-Unit Size
Bandage, compress, Telfa, 3 in., 2 per unit	11290	1	1
Bandage, gauze, 4 in. x 6 yds.	11240	1	1
Bandage, triangular, 40 in. wide	11160	2	2
Blanket, rescue, 56 in. x 84 in.	11392	0	1
Calomine ointment, 1/8 oz. tube, pkg. of 6 tubes	11120	1	1
Cold Pack	11220	1	1
Compress, 24 in. x 72 in.	11250	1	2
Compress, plastic, Telfa, 16 to pkg.	11210	2	3
Creosote burn wash, 6 vials per unit	11050	0	1
Eye dressing kit, 1 oz. bottle eye wash solution, 2 eye dressing pads, 2 sets adhesive strips	11260	1	1
Forceps and scissors	11230	1	1
Plaster, adhesive, 1/2 in. x 2-1/2 yds., 2 spools per pkg.	11270	1	1
Soap, anti-bacterial	34405	0	1
Swabs, Betadine (Povidone-iodine), 10 per unit	11060	1	2
Swabs, Sting-kill, 10 per box	11130	1	2
First-Aid Instructions Handbook	11090	1	1

## APPENDIX D

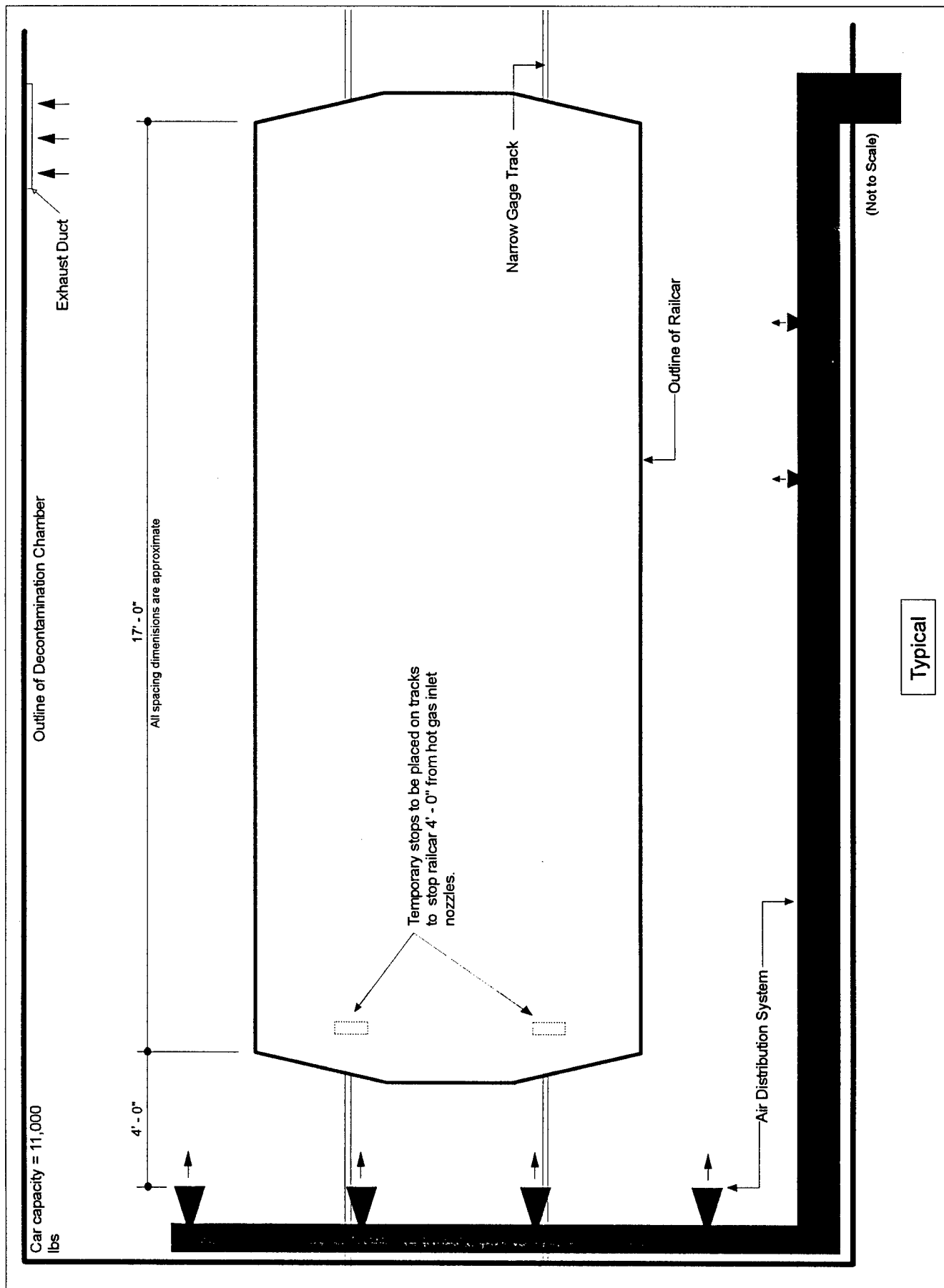


Figure D-1 Railcar Placement Inside Chamber

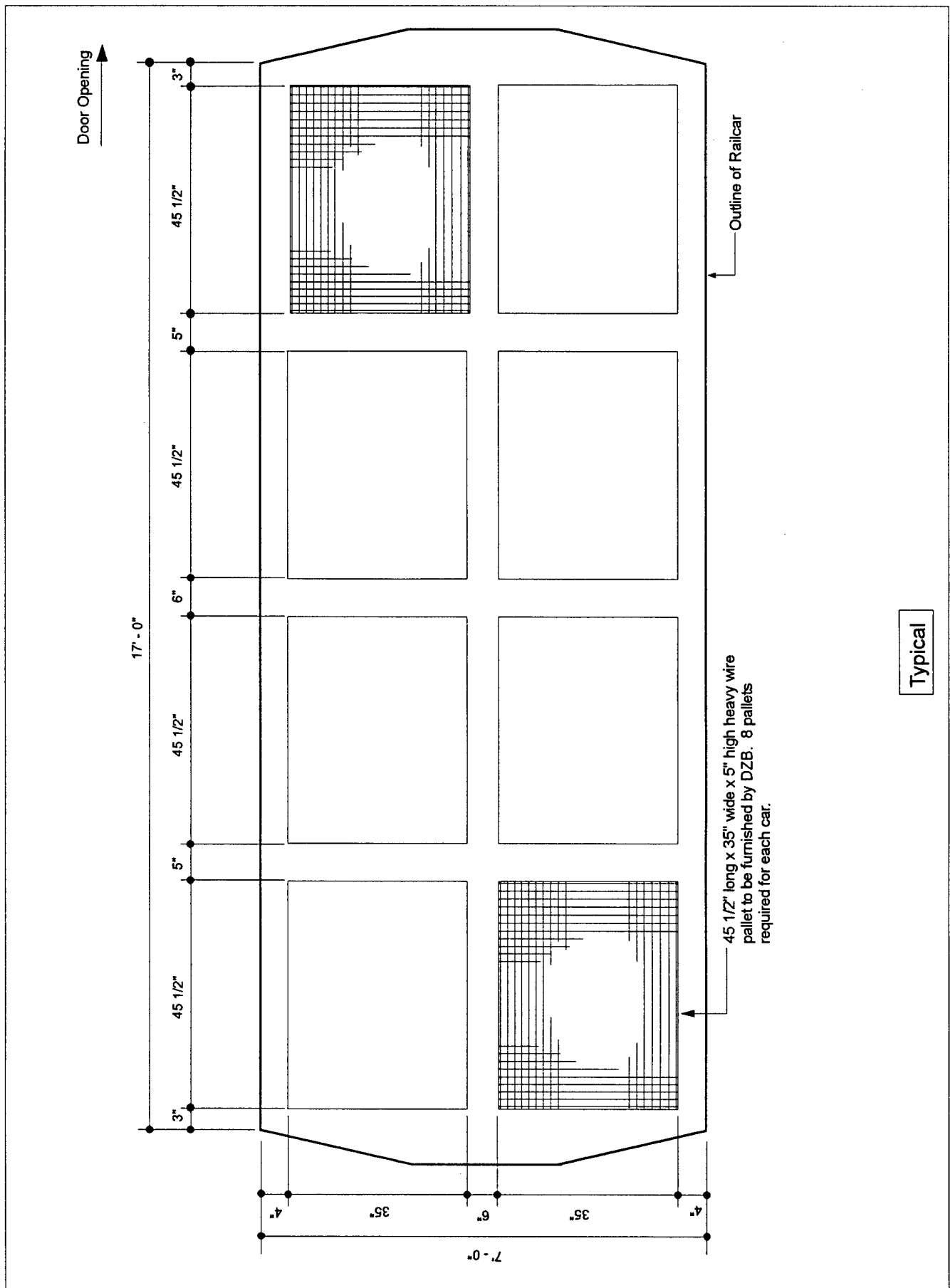
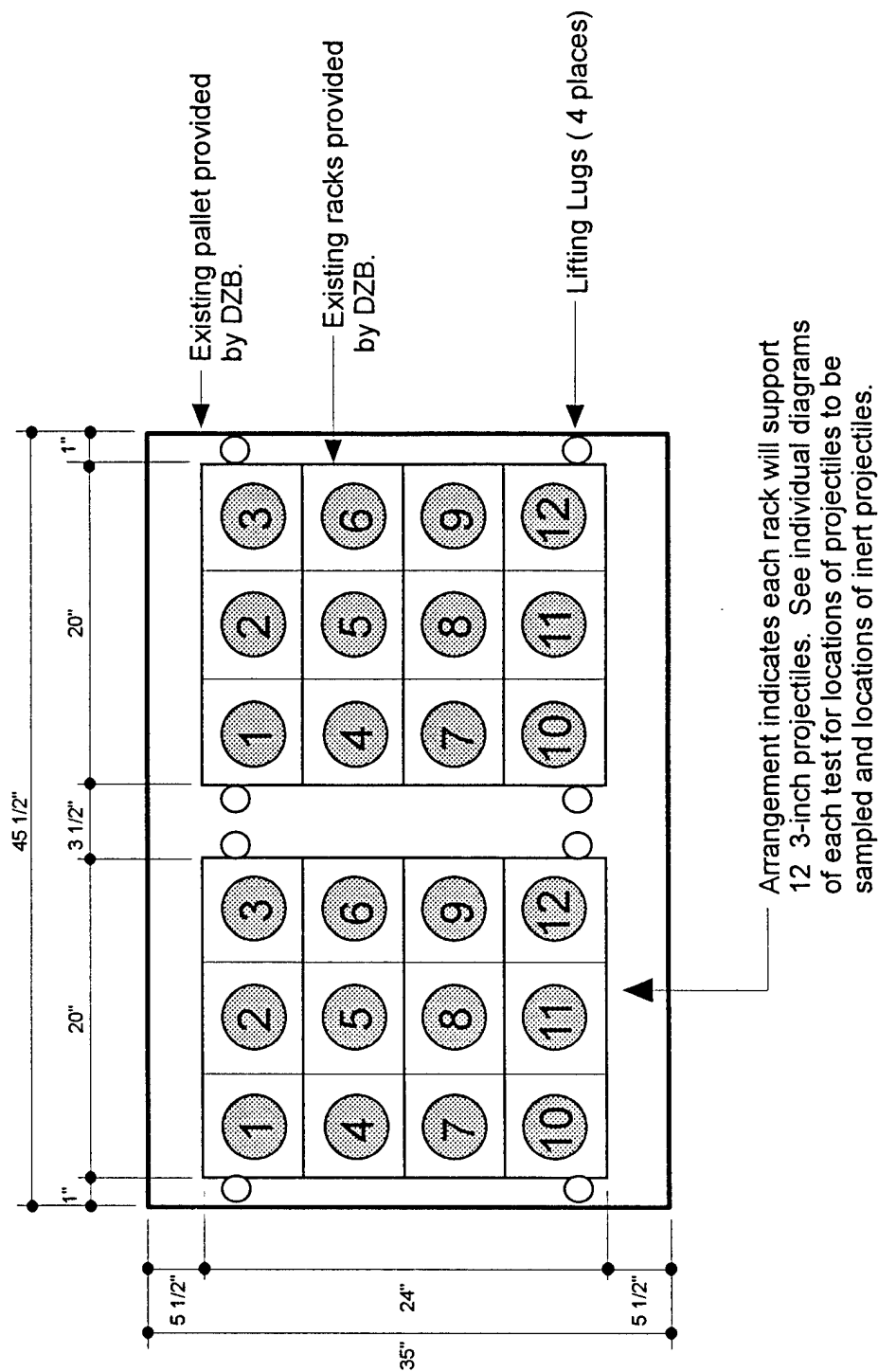


Figure D-2 Typical Pallet Arrangement on Railcar

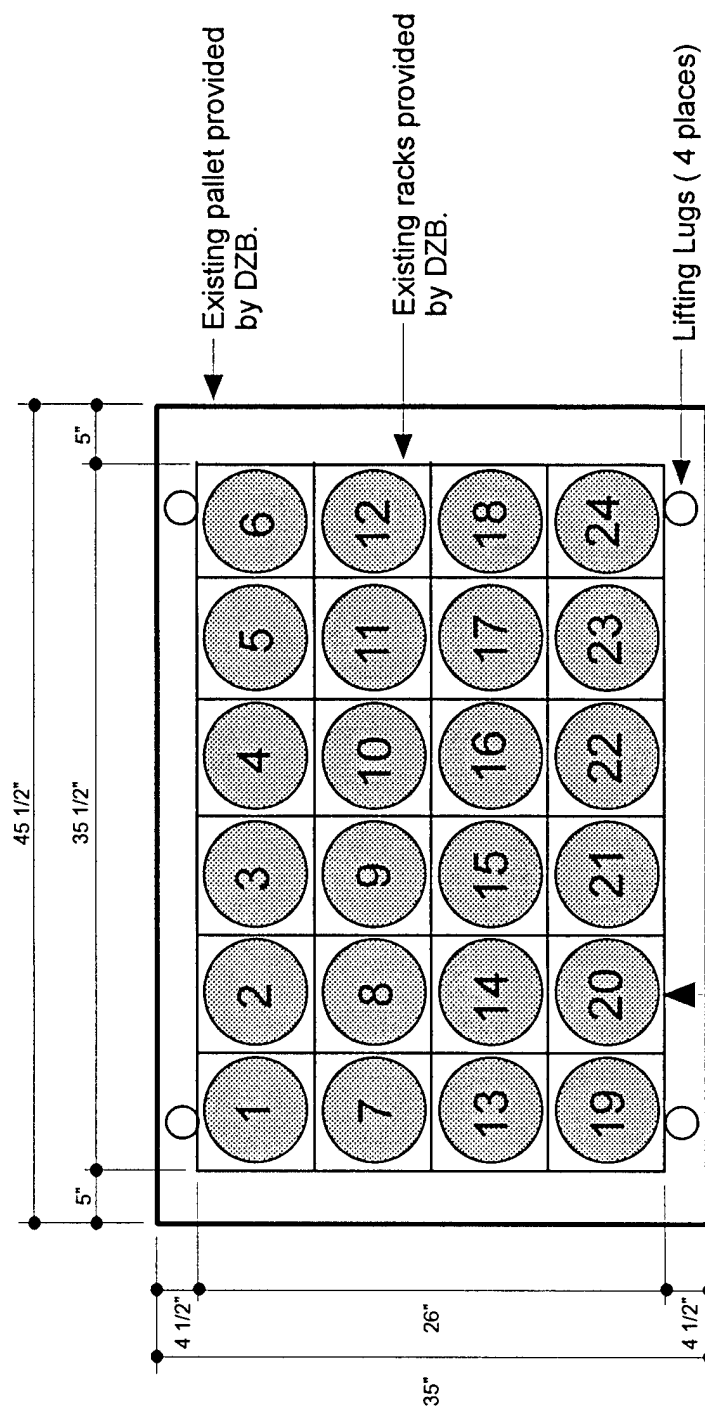
## 3-inch Projectiles



Typical

Figure D-3 3-inch Projectile Racks Arranged on Pallet

# 5-inch Projectiles

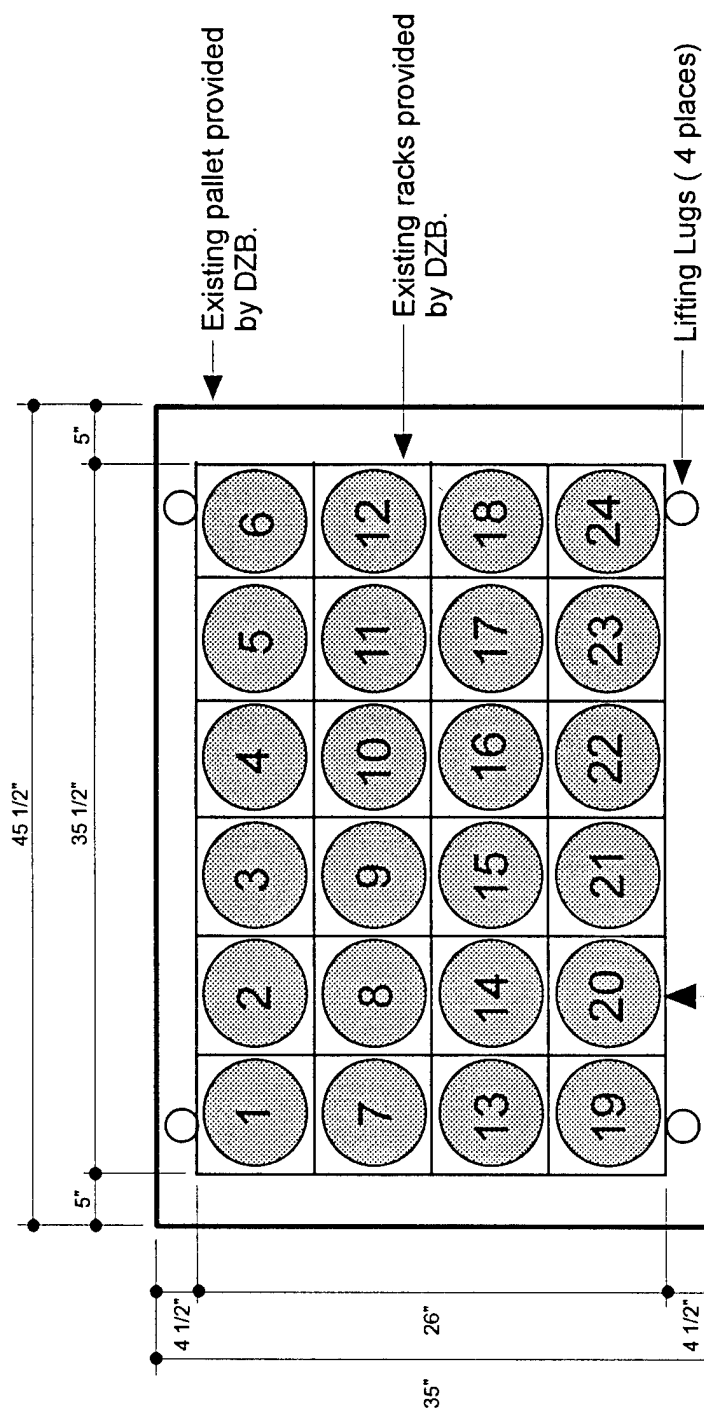


Arrangement indicates each rack will support 24 5-inch projectiles. See individual diagrams of each test for locations of projectiles to be sampled and locations of inert projectiles.

Typical

Figure D-4 5-inch Projectile Rack Arranged on Pallet

# 106mm Projectiles



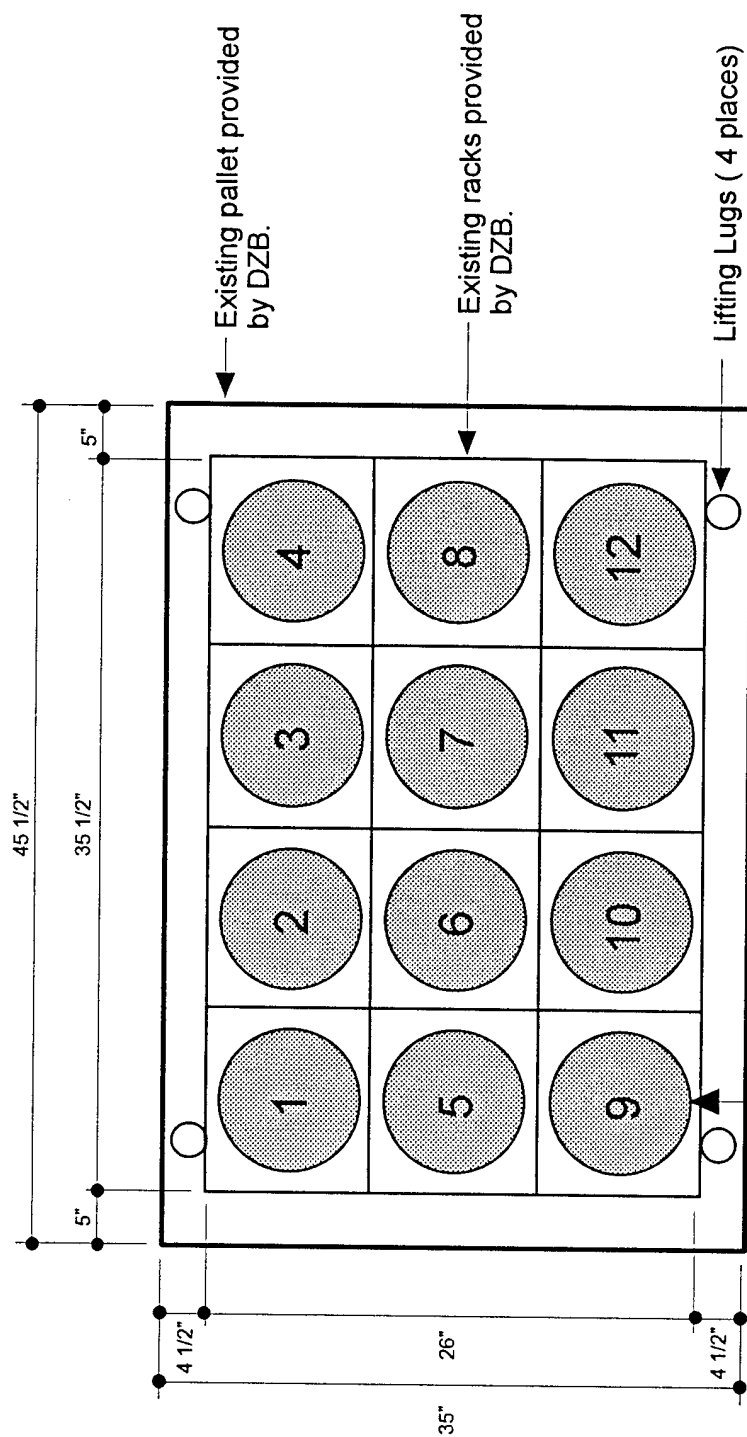
Arrangement indicates each rack will support 24 106mm projectiles. See individual diagrams of each test for locations of projectiles to be sampled and locations of inert projectiles. Use 5-inch racks.

Typical

Figure D-5 106mm Projectile Rack Arranged on Pallet



# 175mm Projectiles

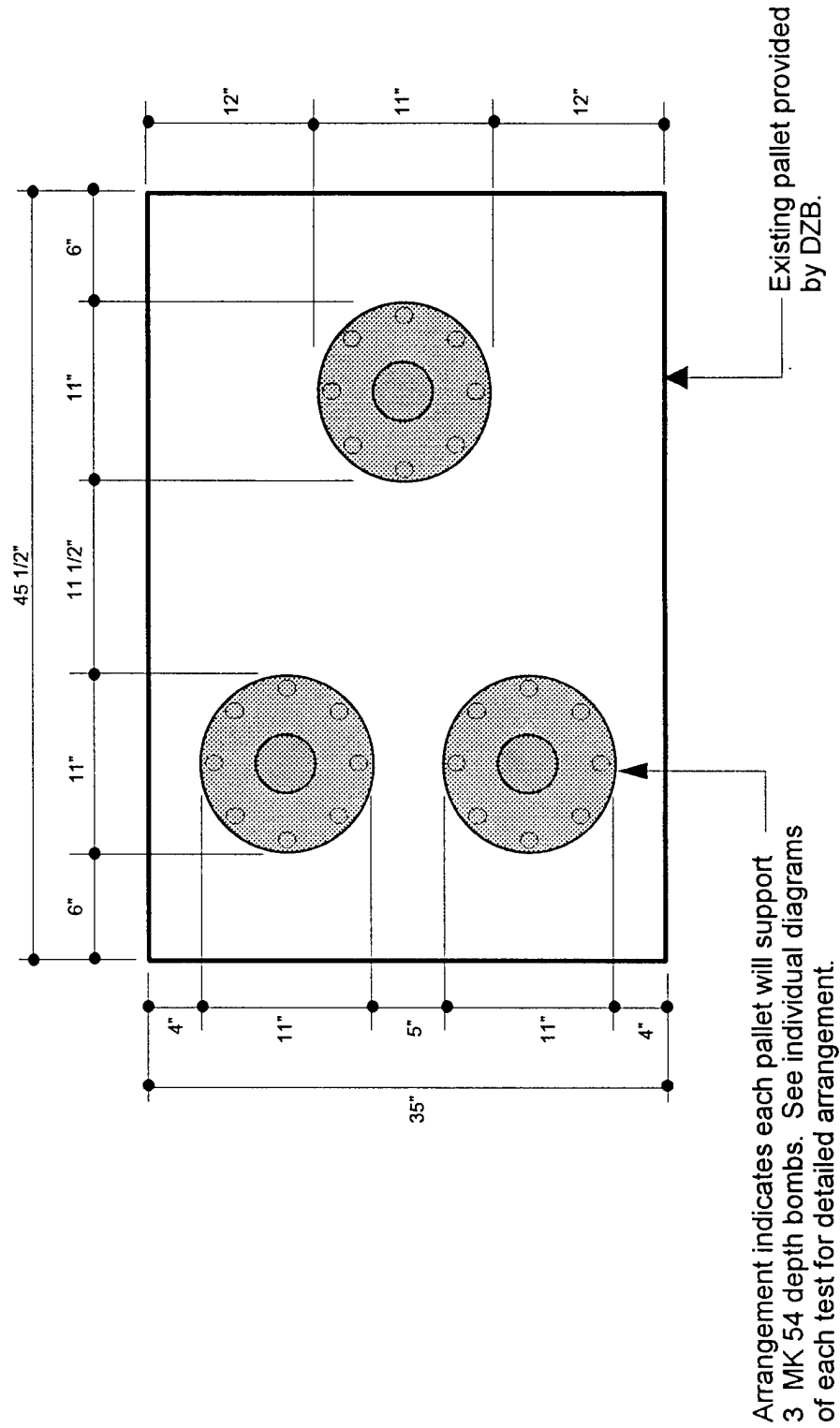


Arrangement indicates each rack will support 12 175mm projectiles. See individual diagrams of each test for locations of projectiles to be sampled and locations of inert projectiles.

Typical

Figure D-6 175mm Projectile Rack Arranged on Pallet

# MK 54 Depth Bombs

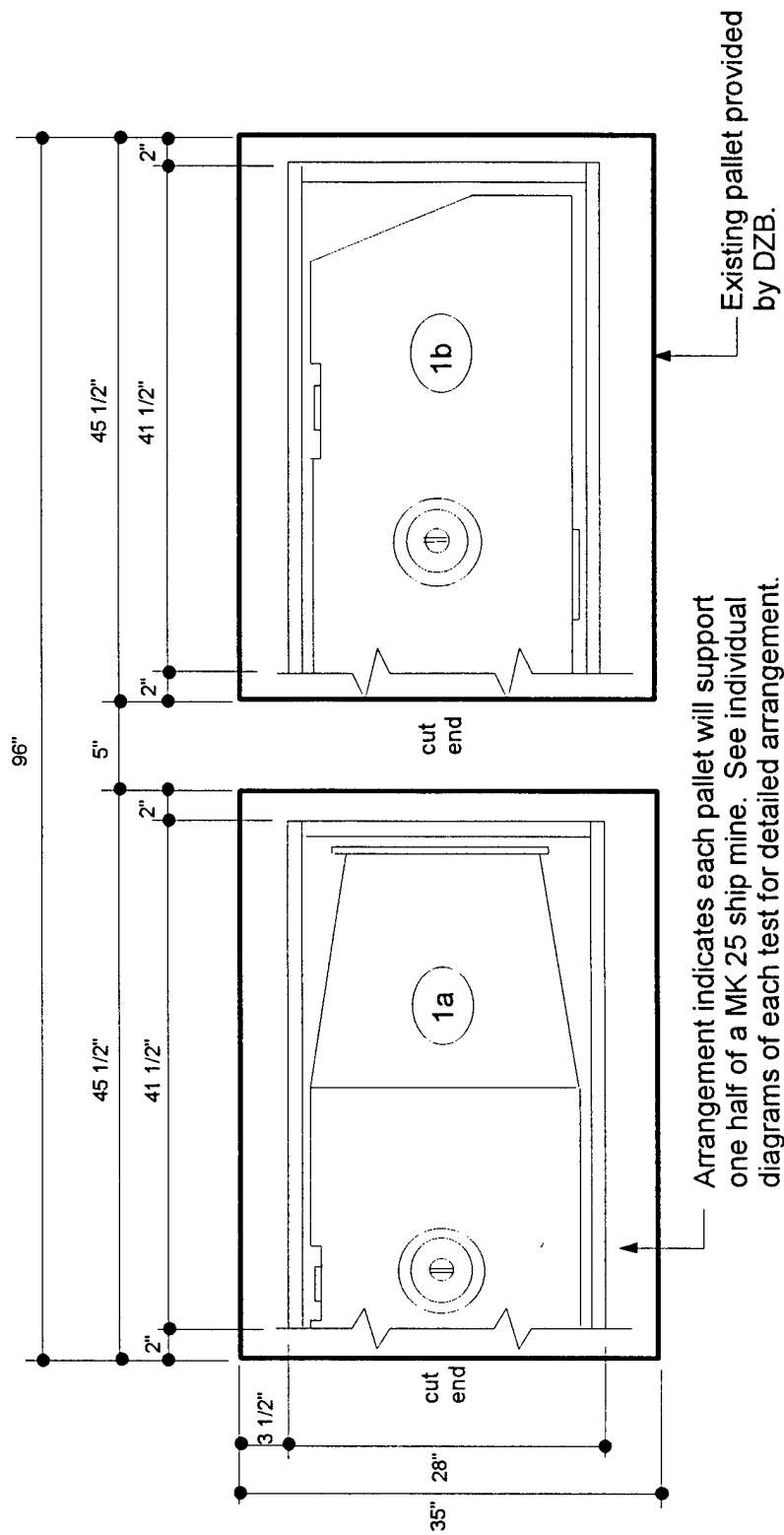


Typical

Figure D-7 MK 54 Depth Bombs (Sawed Ends) Arranged on Pallet

# MK 25 Ship Mines

(Mines cut in half)

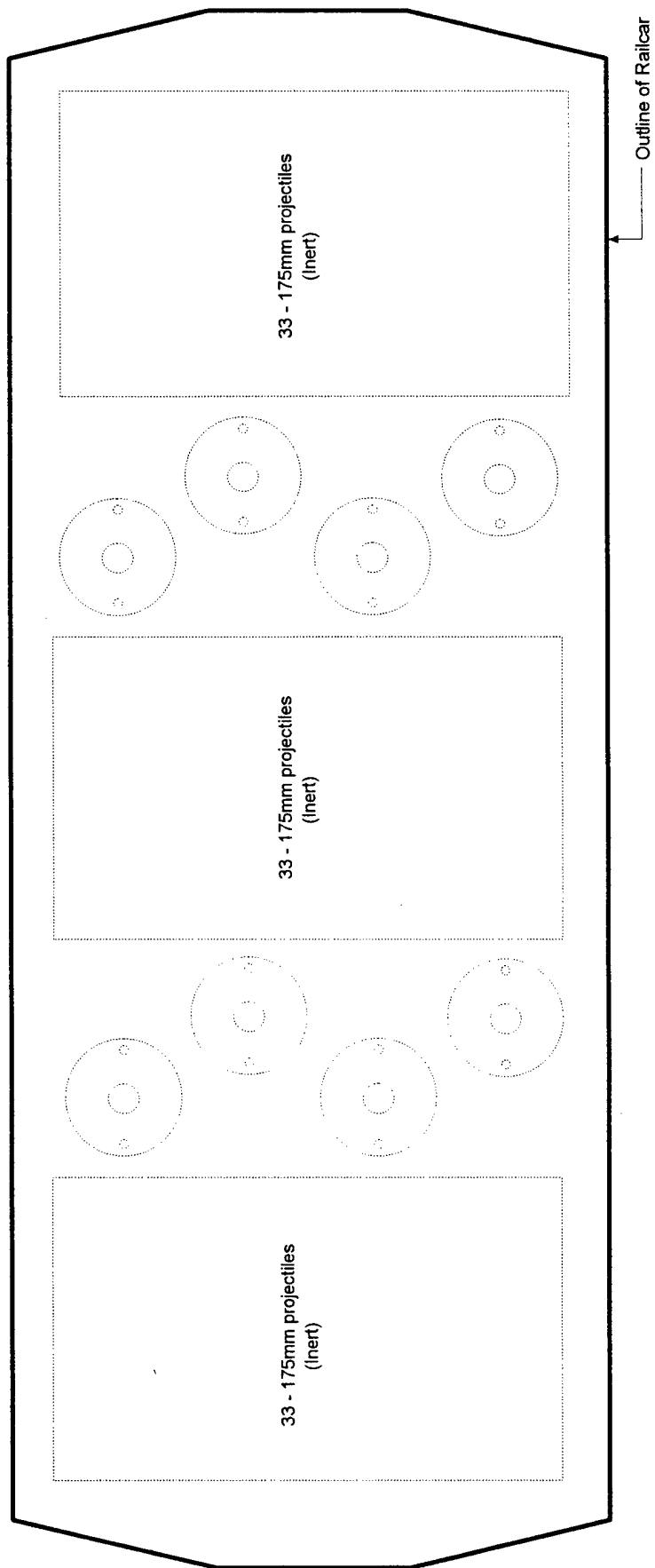


Typical

Figure D-8 MK 25 Ship Mines Arranged on Pallets

Car Capacity = 11,000 lbs.

Door Opening →



This test is discontinued, configuration has been dismantled

Test A

Figure D-9 Rail Car Configuration During Prove-Out Test

Car Capacity = 11,000 lbs.

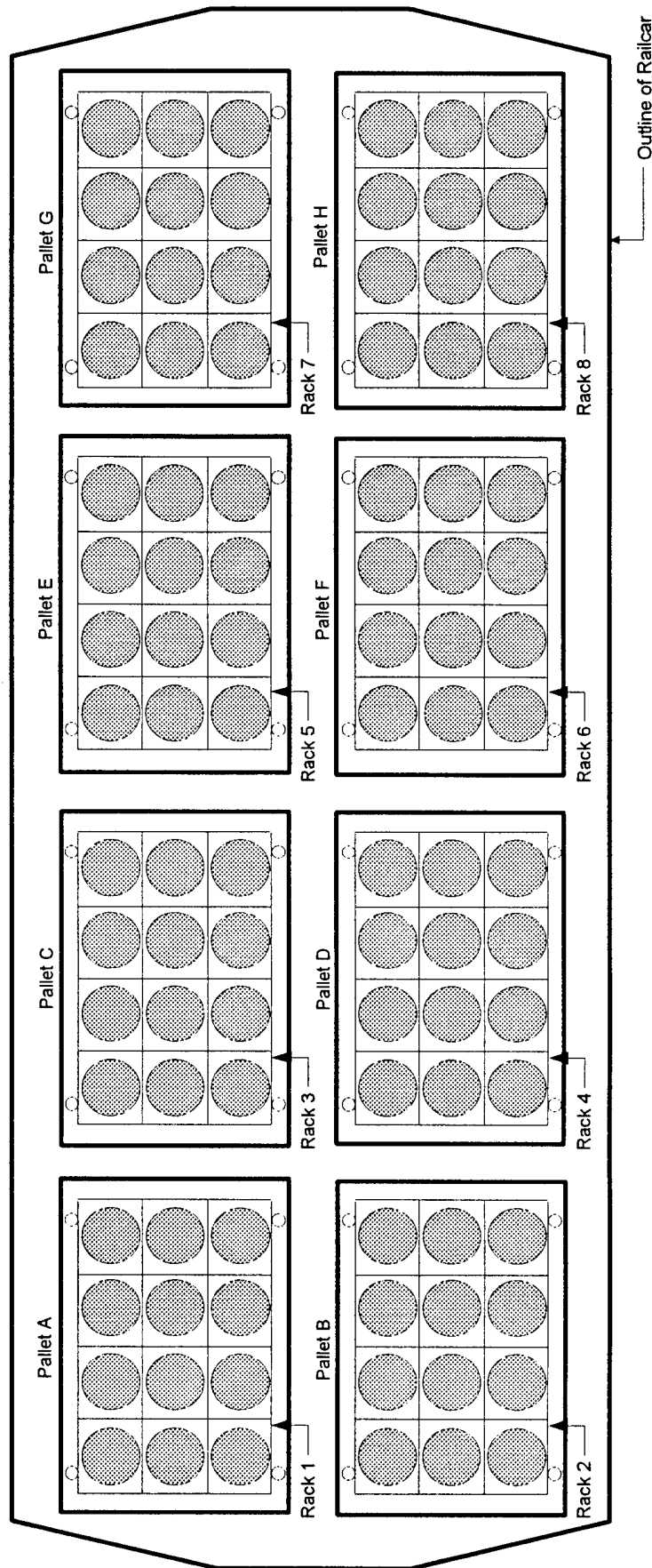
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

## 175mm Projectiles

(Use items from FF-13)

Door Opening



Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test B

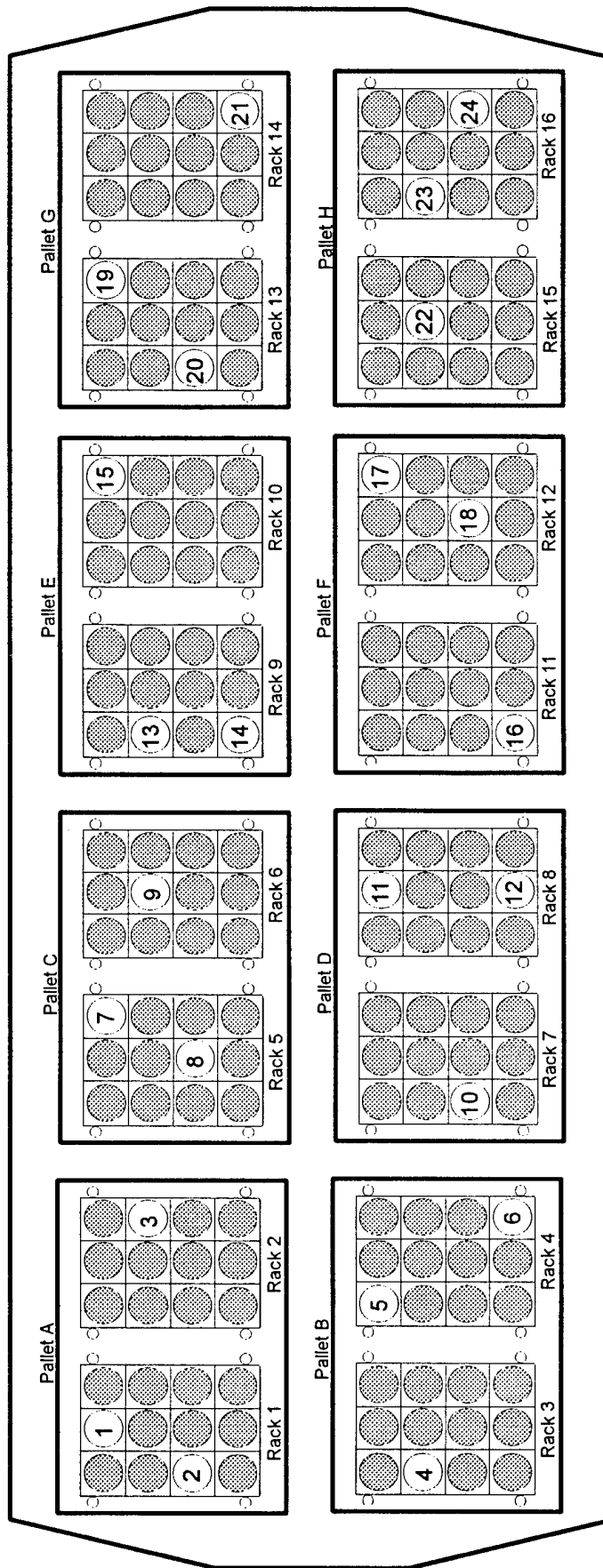
Figure D-10 175mm Projectiles (Inert)

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs.  
 Total Weight, 192 projectiles = 1,728 lbs.

## 3" Projectiles

(Use items from FF-13)

Door Opening →



④ Spiked 3" projectile to be sampled

● Inert 3" projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for rack placement on pallets

Test 1

6 Hours at 500 degrees F

Figure D-11 3" Projectiles Spiked with TNT

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

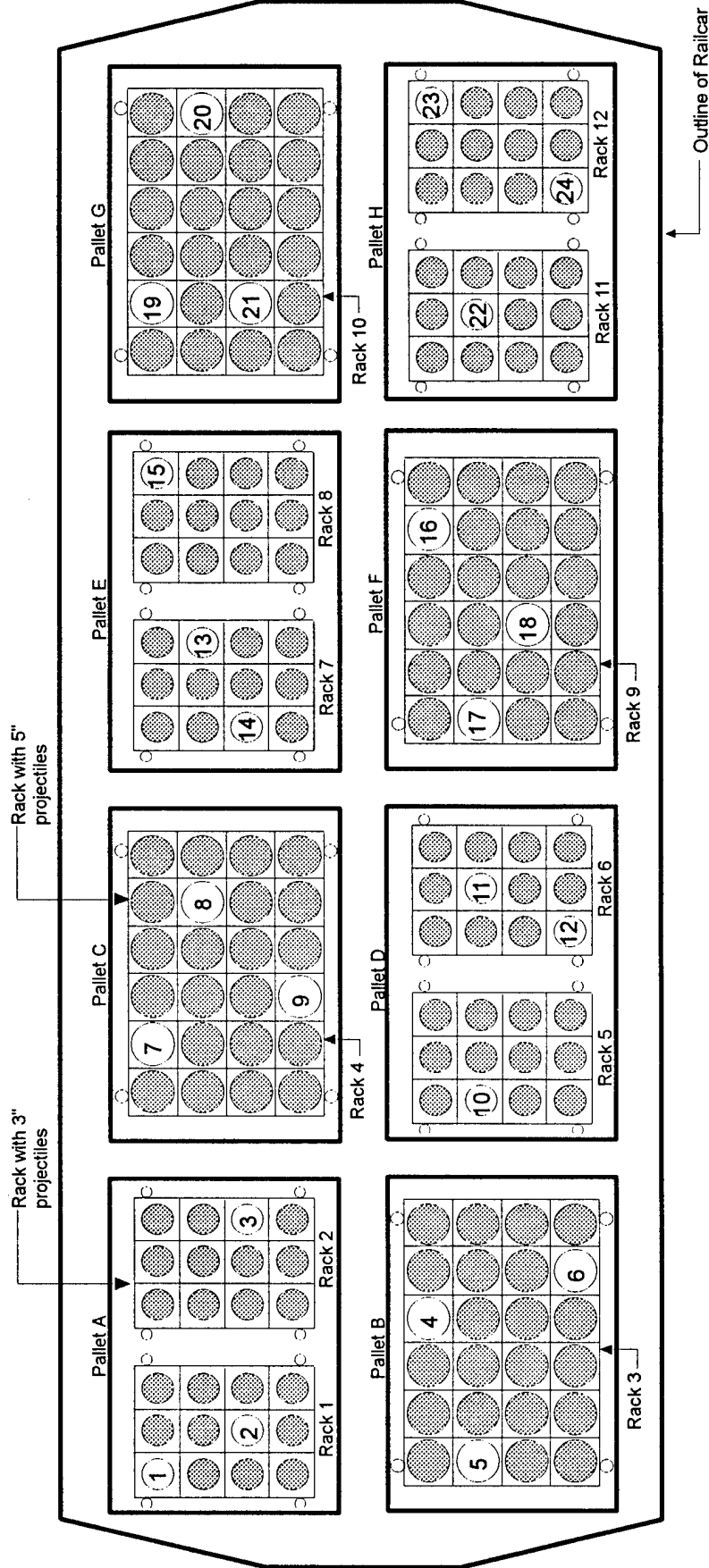
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



- 8 Spiked projectile to be sampled
- Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-3 for 3" rack placement on pallets  
See Figure D-4 for 5" rack placement on pallets

Test 2

6 Hours at 500 degrees F

Figure D-12 3"/5" Projectiles Spiked with RDX

Car Capacity = 11,000 lbs.

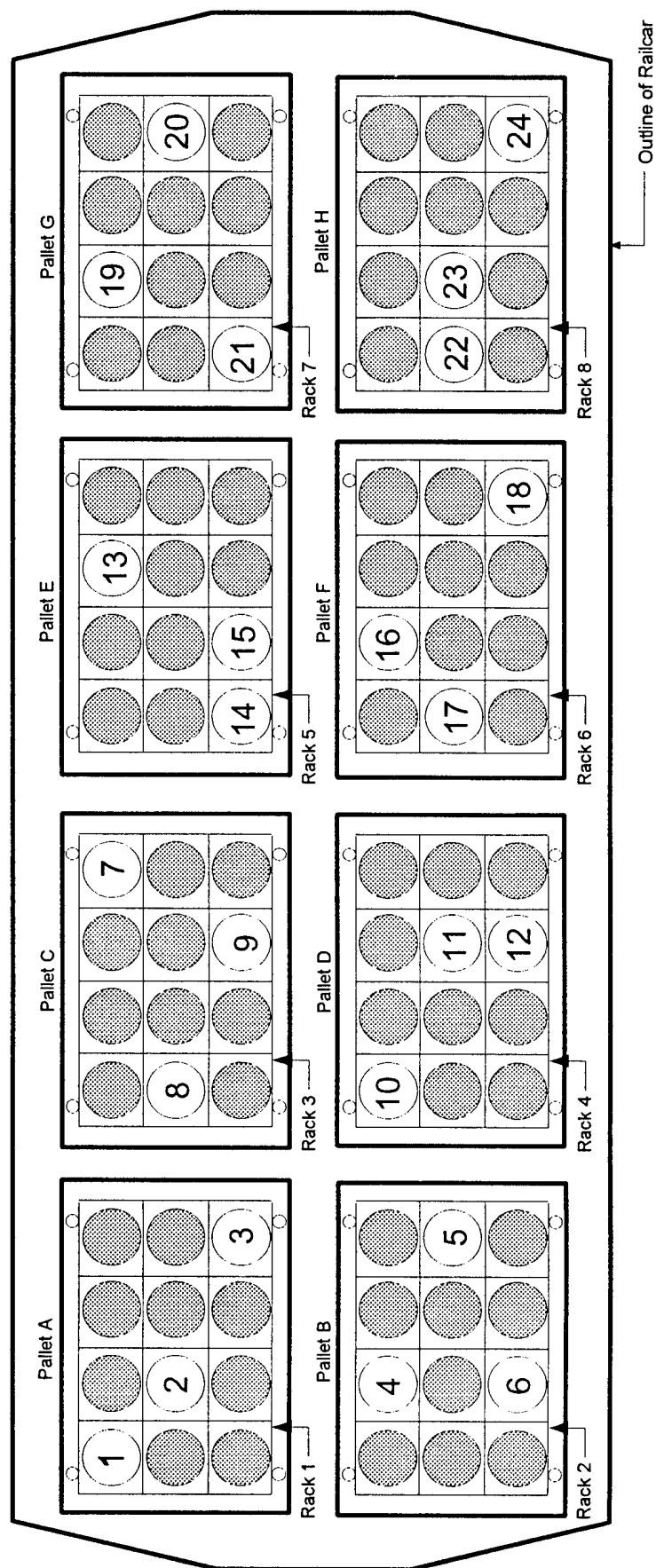
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

## 175mm Projectiles

(Use items from FF-13)

Door Opening



7 Spiked projectile to be sampled

Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test 3

6 Hours at 500 degrees F

Figure D-13 175mm Projectiles Spiked with Comp B

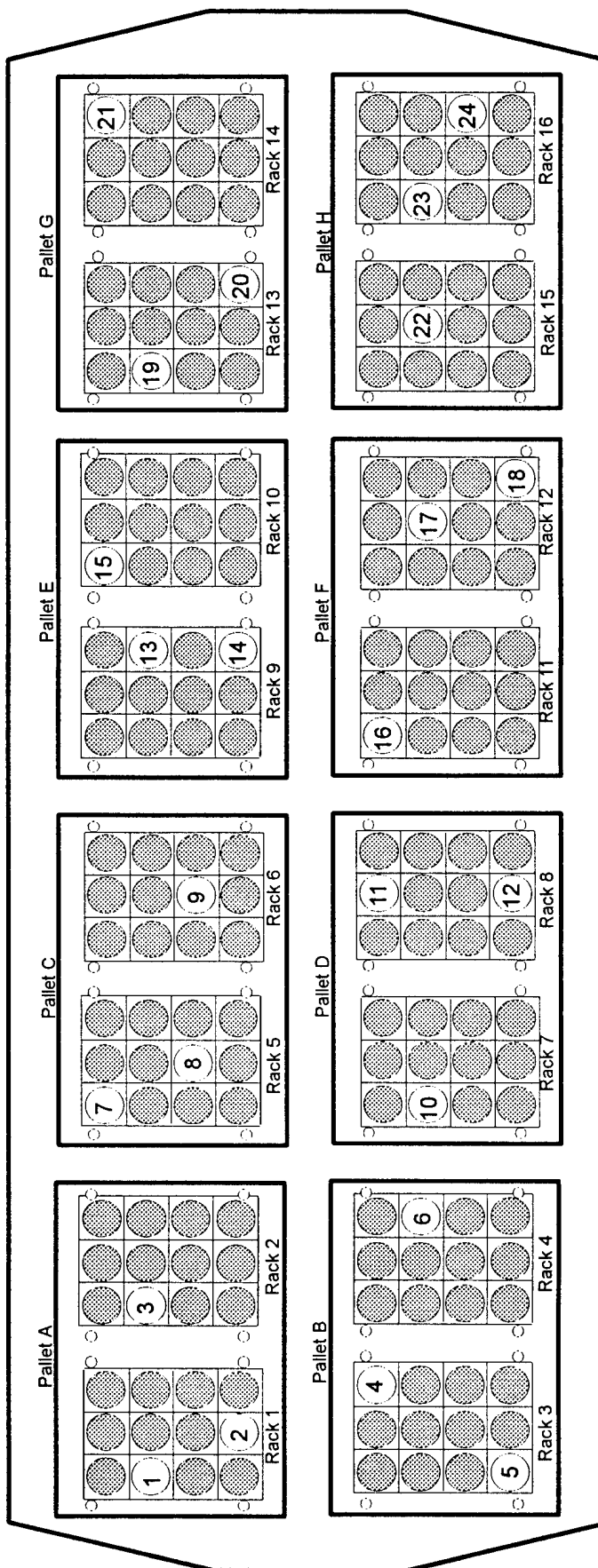


Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs.  
 Total Weight, 192 projectiles = 1,728 lbs.

## 3" Projectiles

(Use items from FF-13)

Door Opening →



④ Spiked 3" projectile to be sampled

● Inert 3" projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for rack placement on pallets

Test 4

6 Hours at 550 degrees F

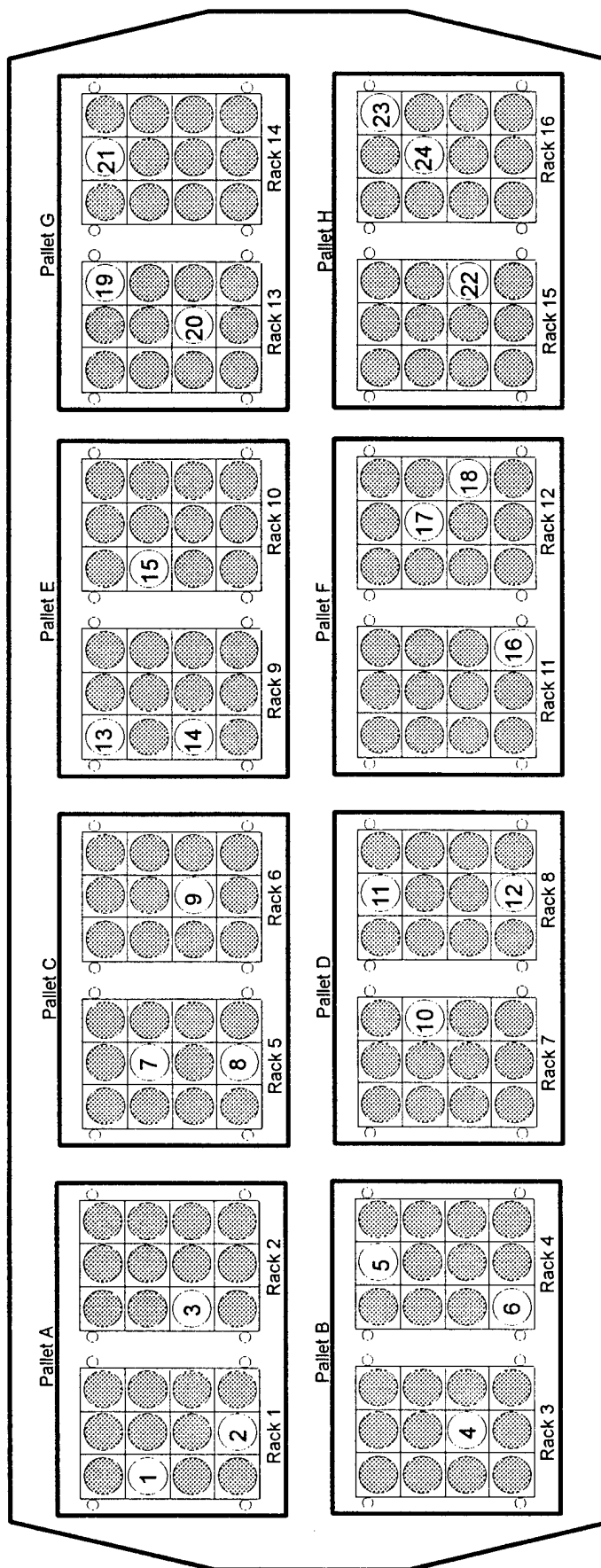
Figure D-14 3" Projectiles Spiked with TNT

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs.  
 Total Weight, 192 projectiles = 1,728 lbs.

## 3" Projectiles

(Use items from FF-13)

Door Opening



5 Spiked 3" projectile to be sampled

Inert 3" projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for rack placement on pallets

Test 5

8 Hours at 500 degrees F

Figure D-15 3" Projectiles Spiked with HBX

Car Capacity = 11,000 lbs.

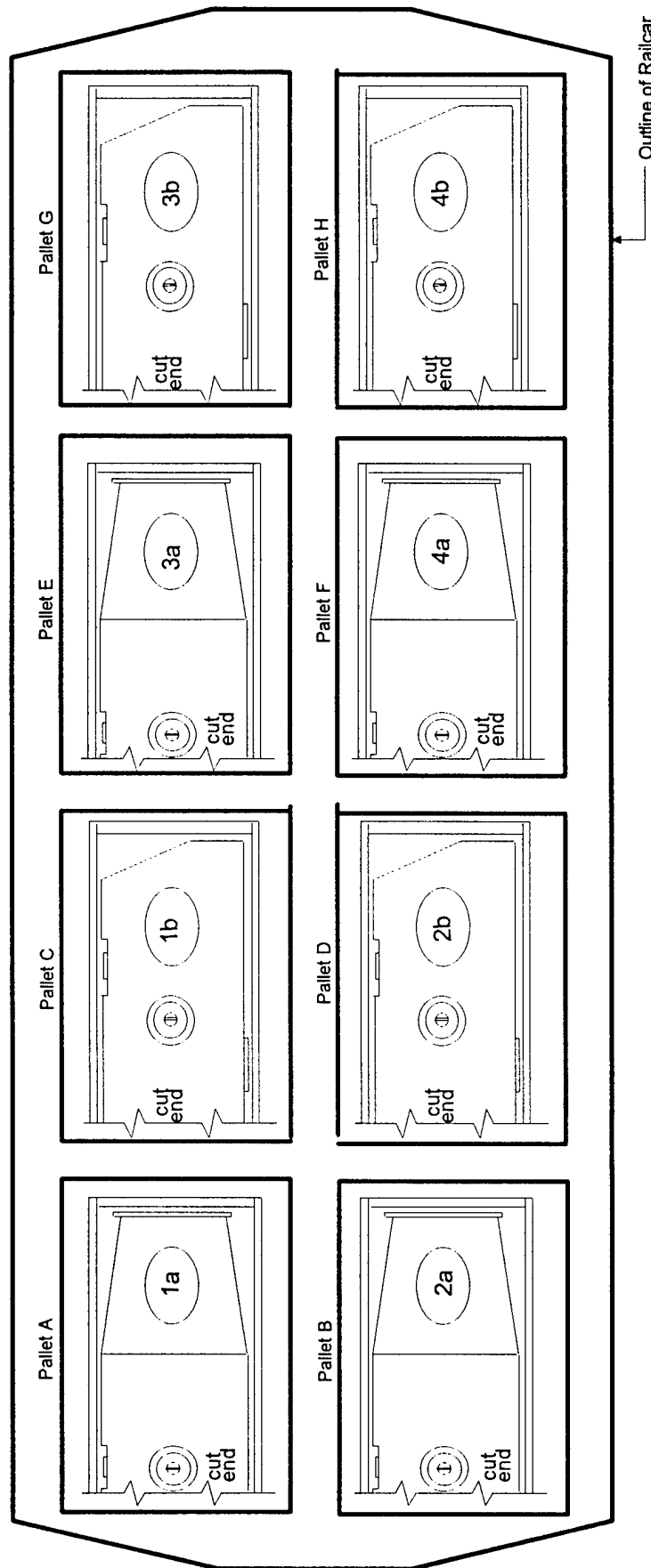
Unit Weight, MK 25 = 715 lbs

Total Weight, 4 Mines = 2,860 lbs

## MK 25 Ship Mines

(Unused mines - internals coated with hot-melt)

Door Opening



1a Spiked mine half to be sampled

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-8 for mine placement on pallets

Test 6

16 Hours at 750 degrees F

Figure D-16 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

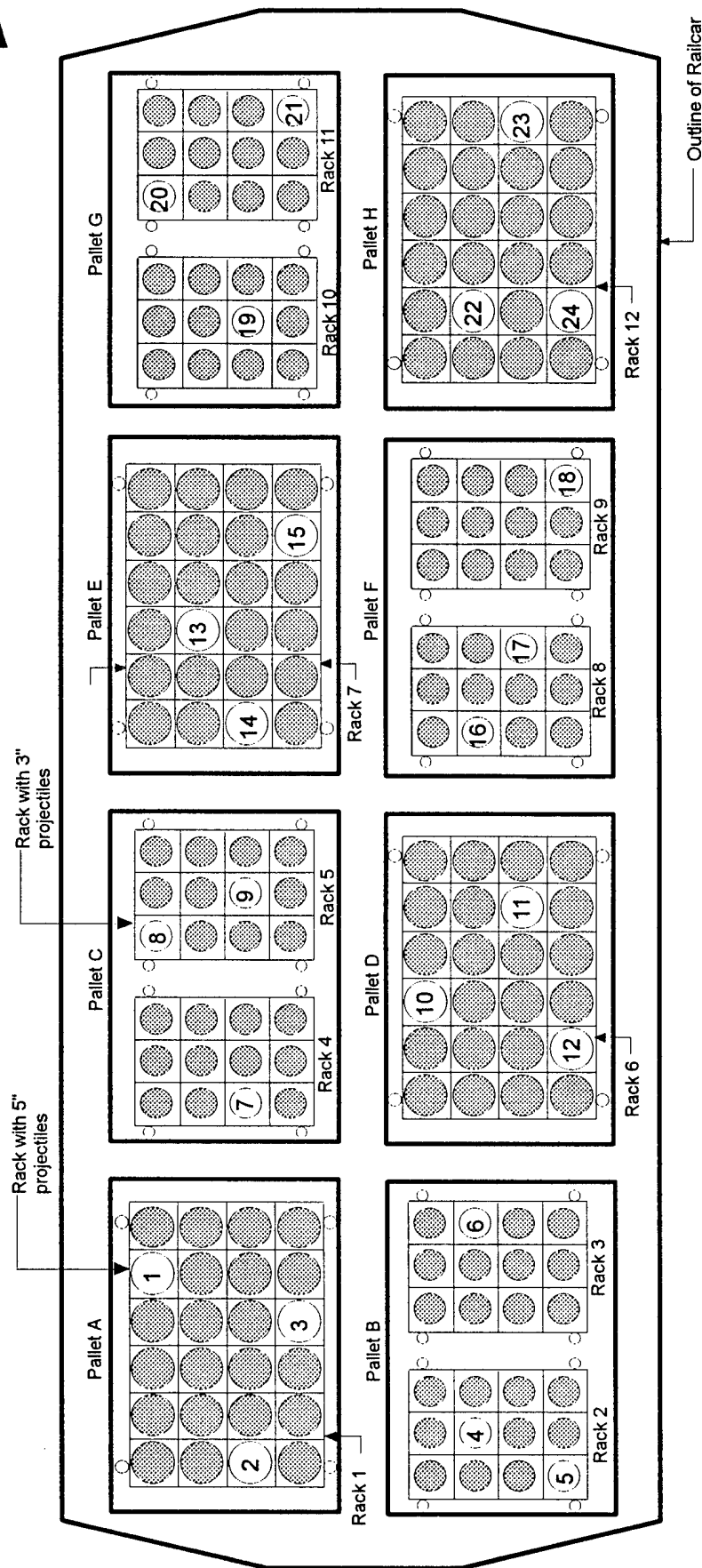
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



Test 7

Base on results of Test 2

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for 3" rack placement on pallets  
 See Figure D-4 for 5" rack placement on pallets

Figure D-17 3"/5" Projectiles Spiked with RDX

Car Capacity = 11,000 lbs.

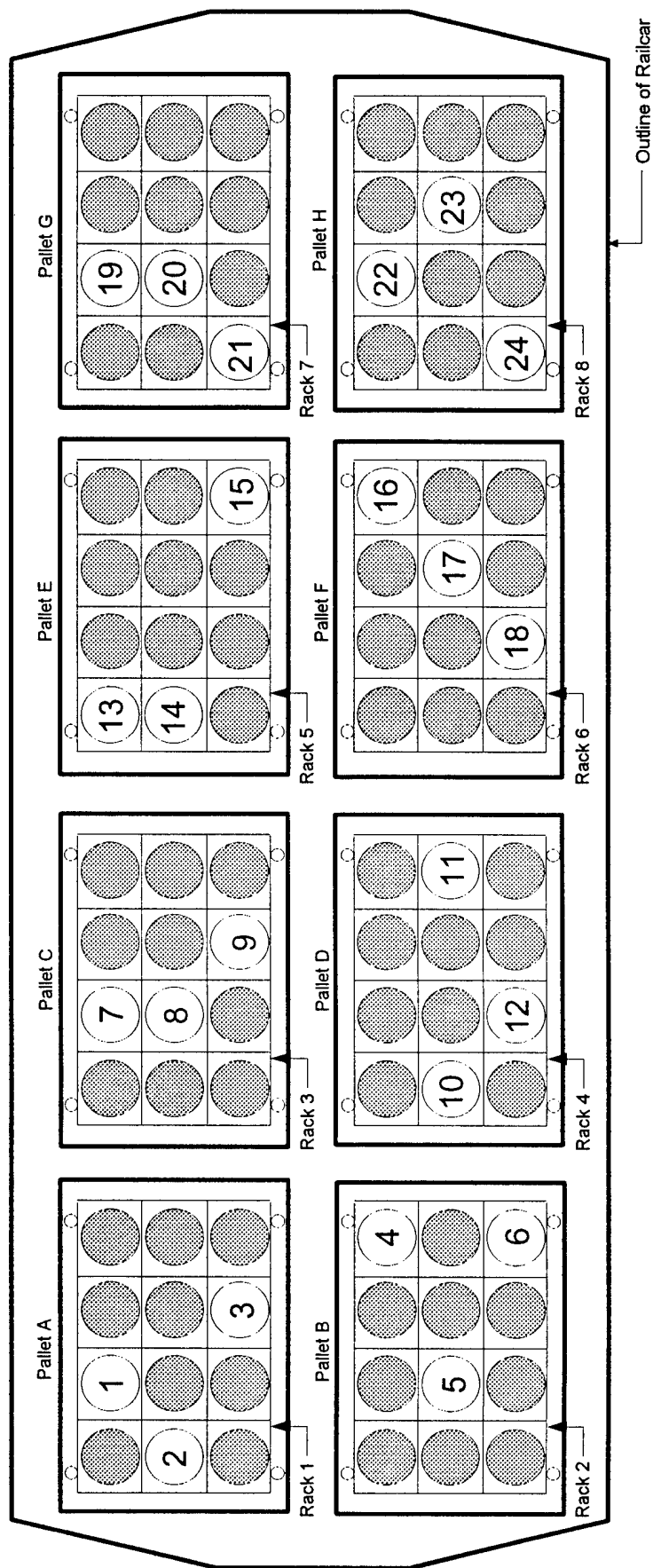
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

# 175mm Projectiles

(Use items from FF-13)

Door Opening



9 Spiked projectile to be sampled



See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test 8

Based on results of Test 3

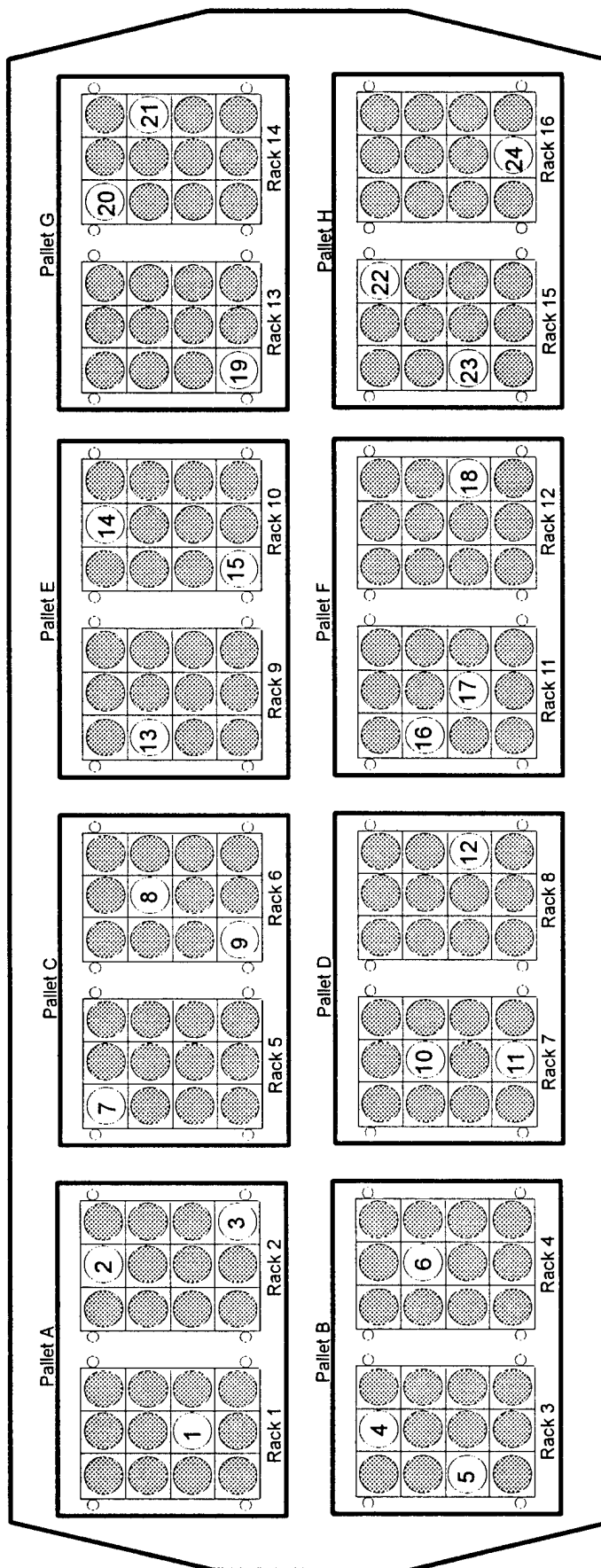
Figure D-18 175mm Projectiles Spiked with Comp B

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs.  
 Total Weight, 192 projectiles = 1,728 lbs.

## 3" Projectiles

(Use items from FF-13)

Door Opening



5 Spiked 3" projectile to be sampled

Inert 3" projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for rack placement on pallets

Test 9

Based on results of Test 4

Figure D-19 3" Projectiles Spiked with HBX

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

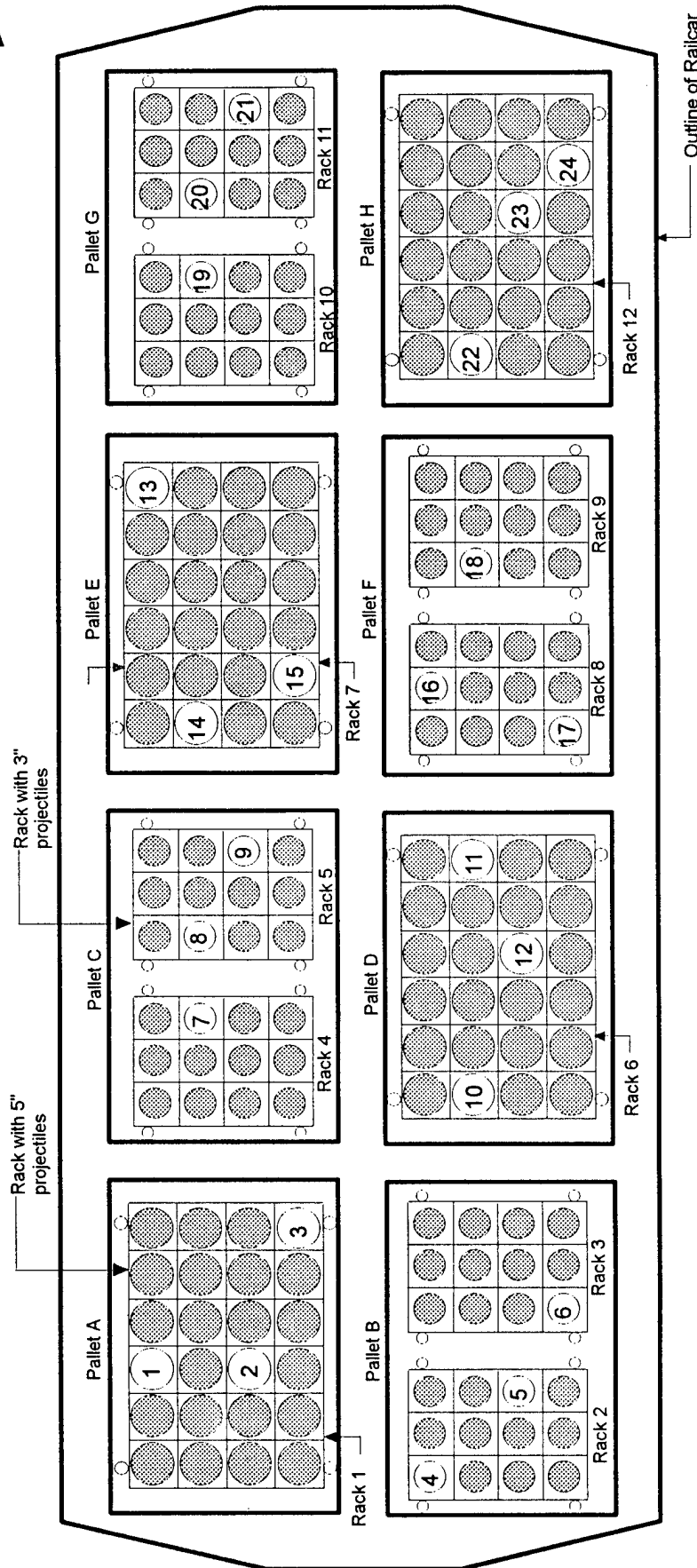
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



9 Spiked projectile to be sampled

● Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-3 for 3" rack placement on pallets

See Figure D-4 for 5" rack placement on pallets

Test 10

12 Hours at 600 degrees F

Figure D-20 3"/5" Projectiles Spiked with Yellow D

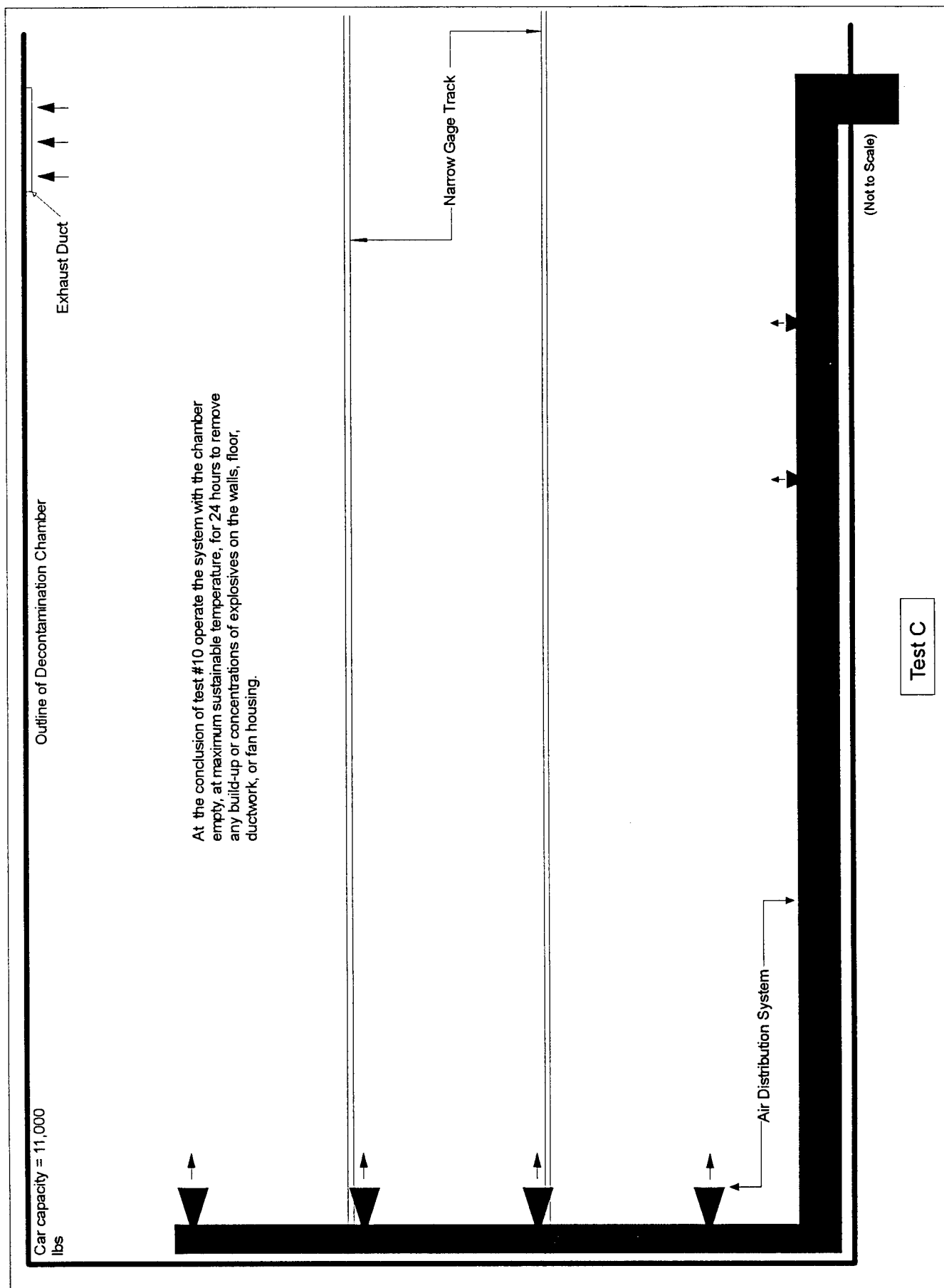


Figure D-21 Empty Chamber Run



Car Capacity = 11,000 lbs.

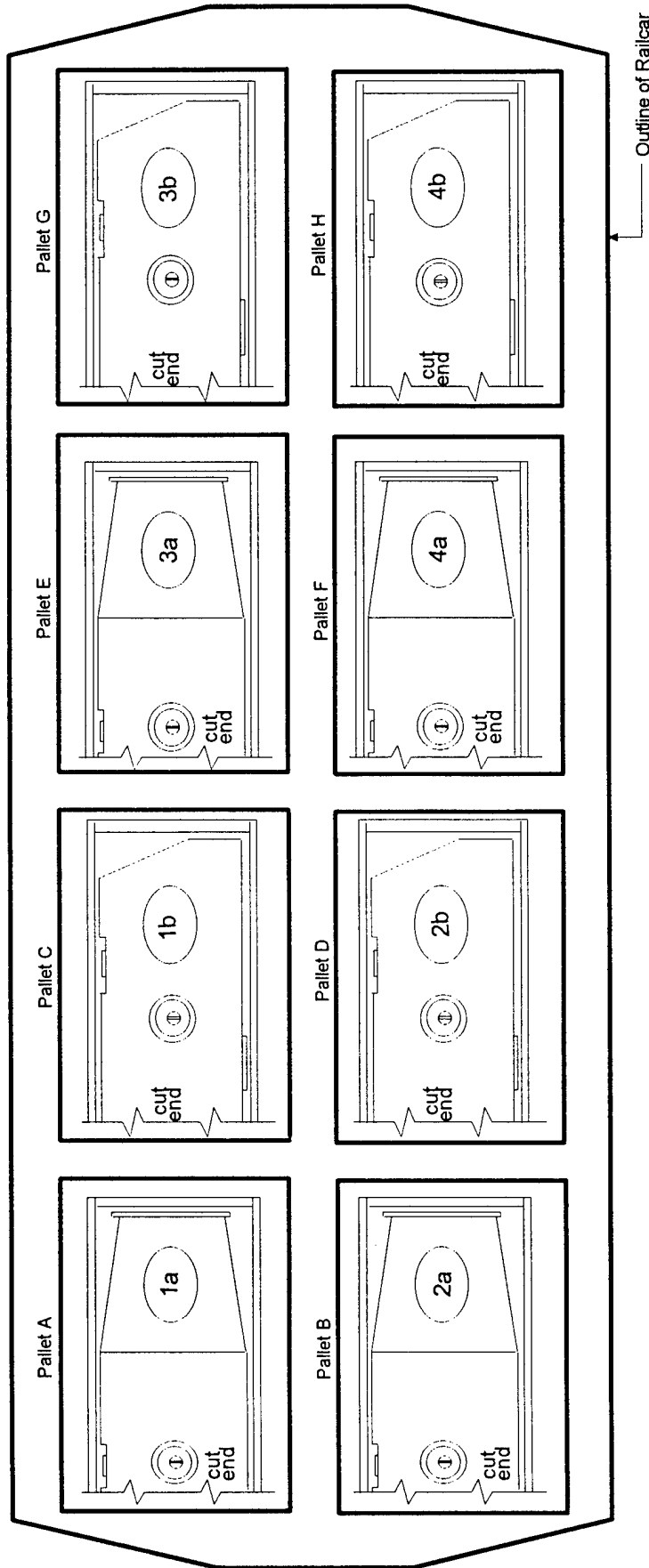
Unit Weight, MK 25 = 715 lbs

Total Weight, 4 Mines = 2,860 lbs

## MK 25 Ship Mines

(Unused mines - internals coated with hot-melt)

Door Opening



2a Spiked mine half to be sampled

Test 11

Based on results of Test 6

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-8 for mine placement on pallets

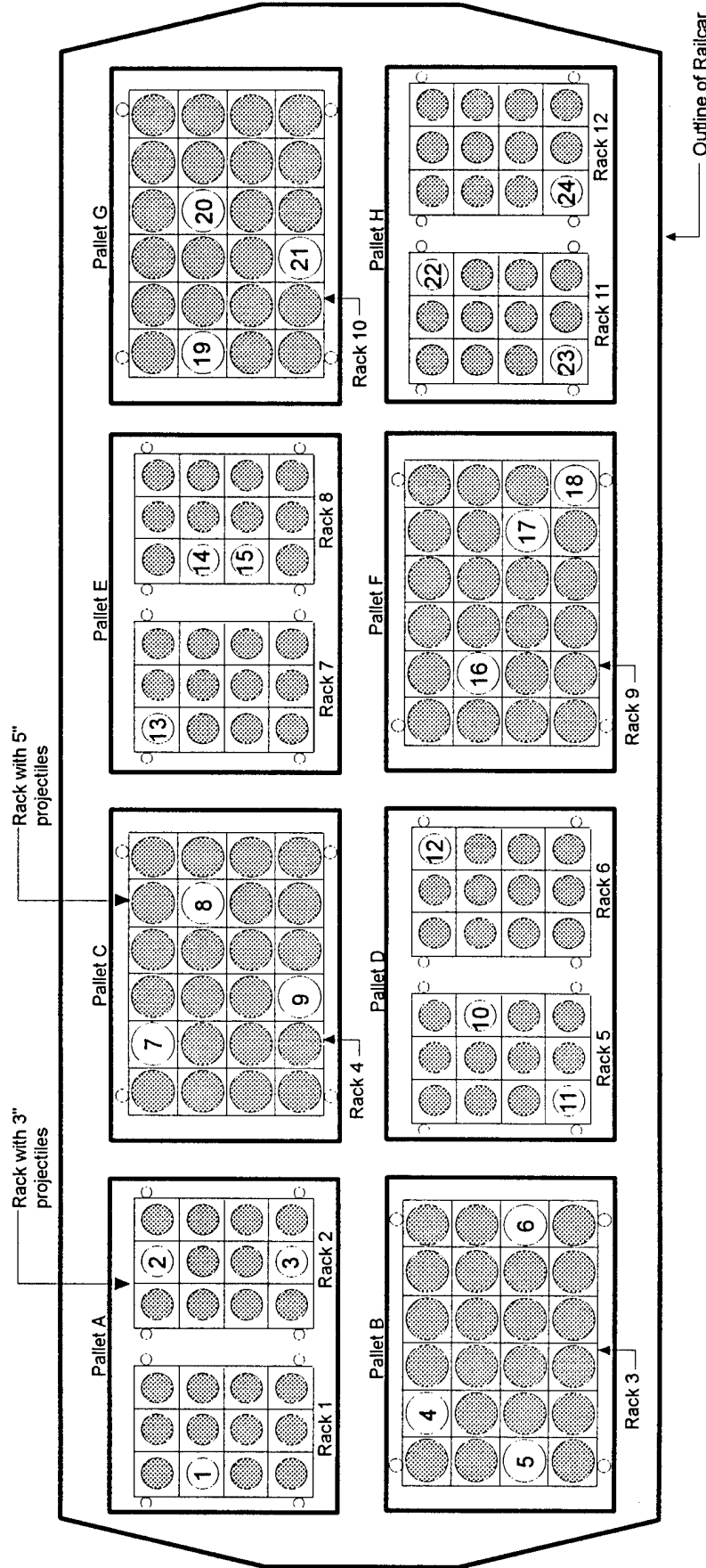
Figure D-22 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

# 3"/5" Projectiles

(Use items from FF-13)

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs  
 Unit Weight, 5" projectile = 67 lbs  
 Total Weight, 192 projectiles = 7,296 lbs

Door Opening



See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for 3" rack placement on pallets  
 See Figure D-4 for 5" rack placement on pallets

Test 12

Based on results of Test 7

Figure D-23 3"/5" Projectiles Spiked with RDX

Car Capacity = 11,000 lbs.

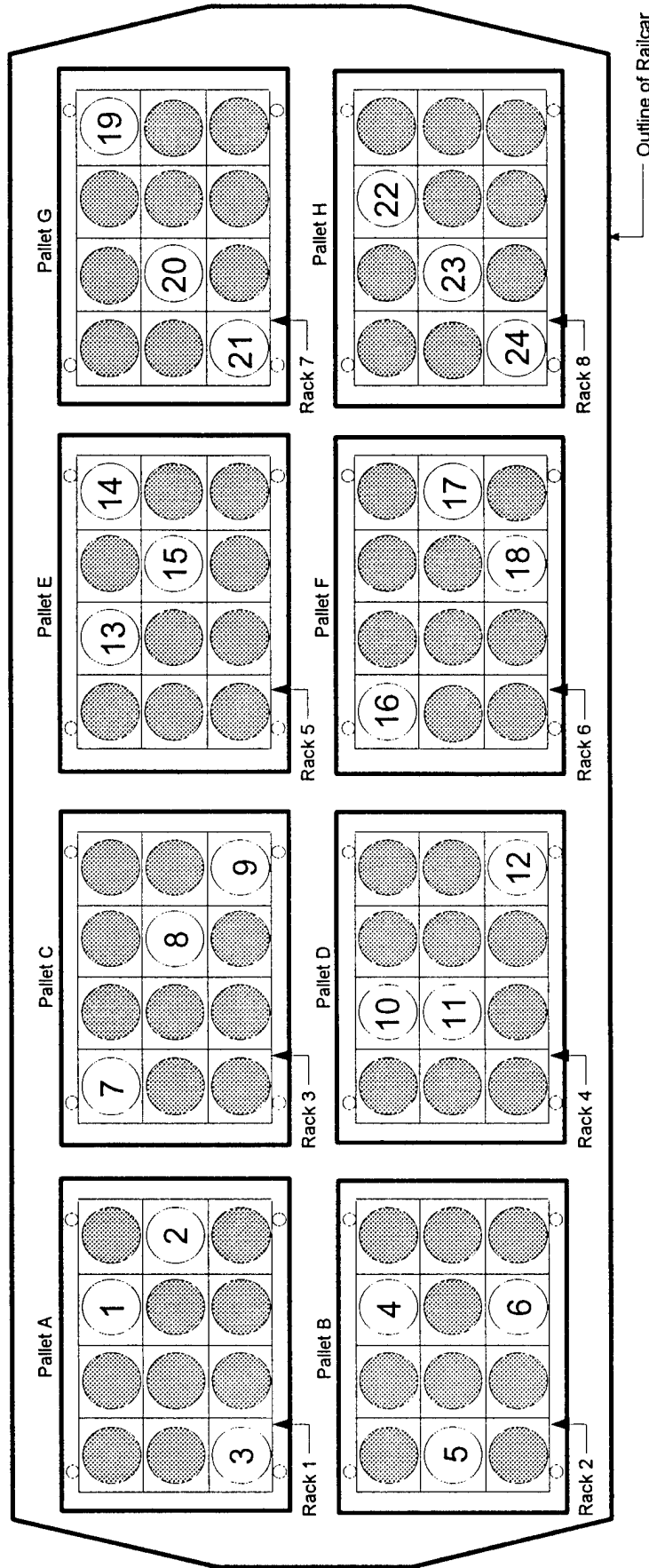
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

## 175mm Projectiles

(Use items from FF-13)

Door Opening



2 Spiked projectile to be sampled



Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-5 for rack placement on pallets

Test 13

Based on results of Test 8

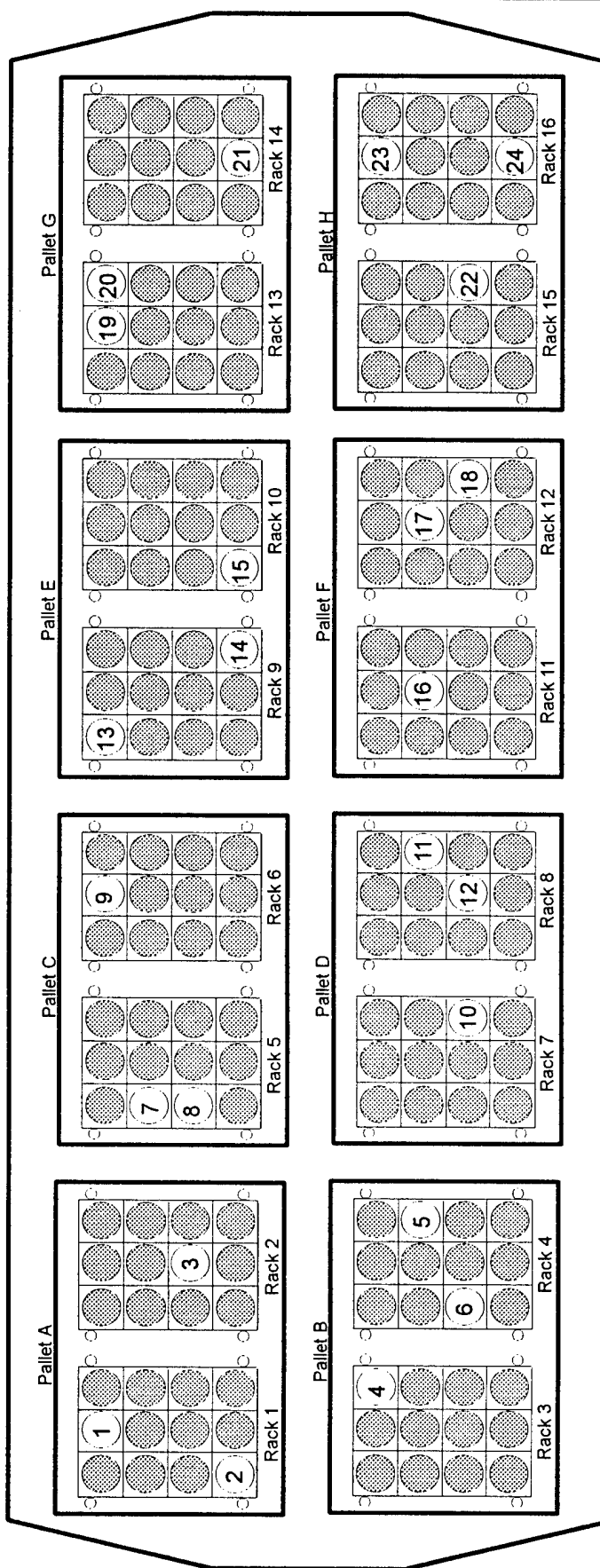
Figure D-24 175mm Projectiles Spiked with Comp B

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs.  
 Total Weight, 192 projectiles = 1,728 lbs.

## 3" Projectiles

(Use items from FF-13)

Door Opening



5 Spiked 3" projectile to be sampled

● Inert 3" projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for rack placement on pallets

Test 14

Based on results of Test 9

Figure D-25 3" Projectiles Spiked with HBX

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

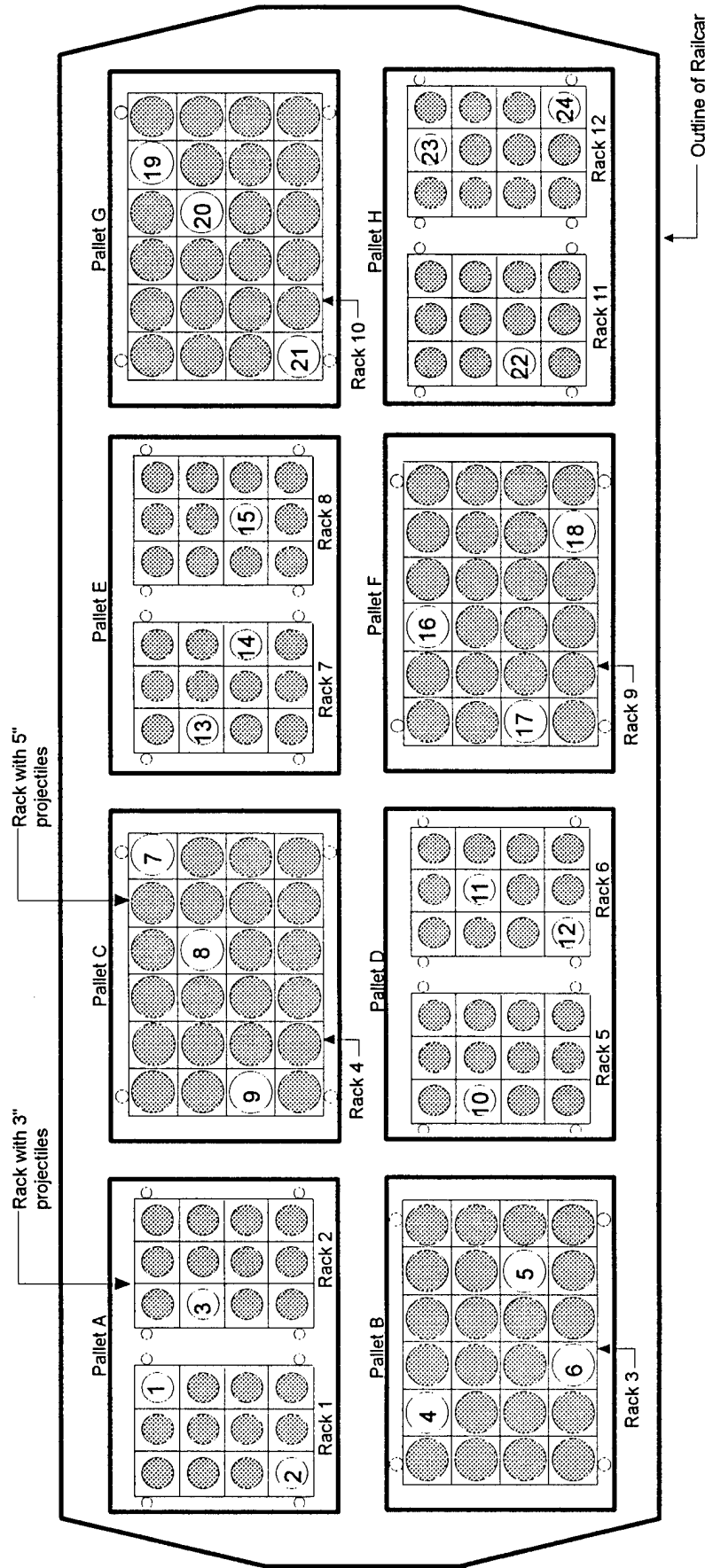
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



Test 15

Based on results of Test 10

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-3 for 3" rack placement on pallets  
See Figure D-4 for 5" rack placement on pallets

Figure D-26 3"/5" Projectiles Spiked with Yellow D

Car Capacity = 11,000 lbs.

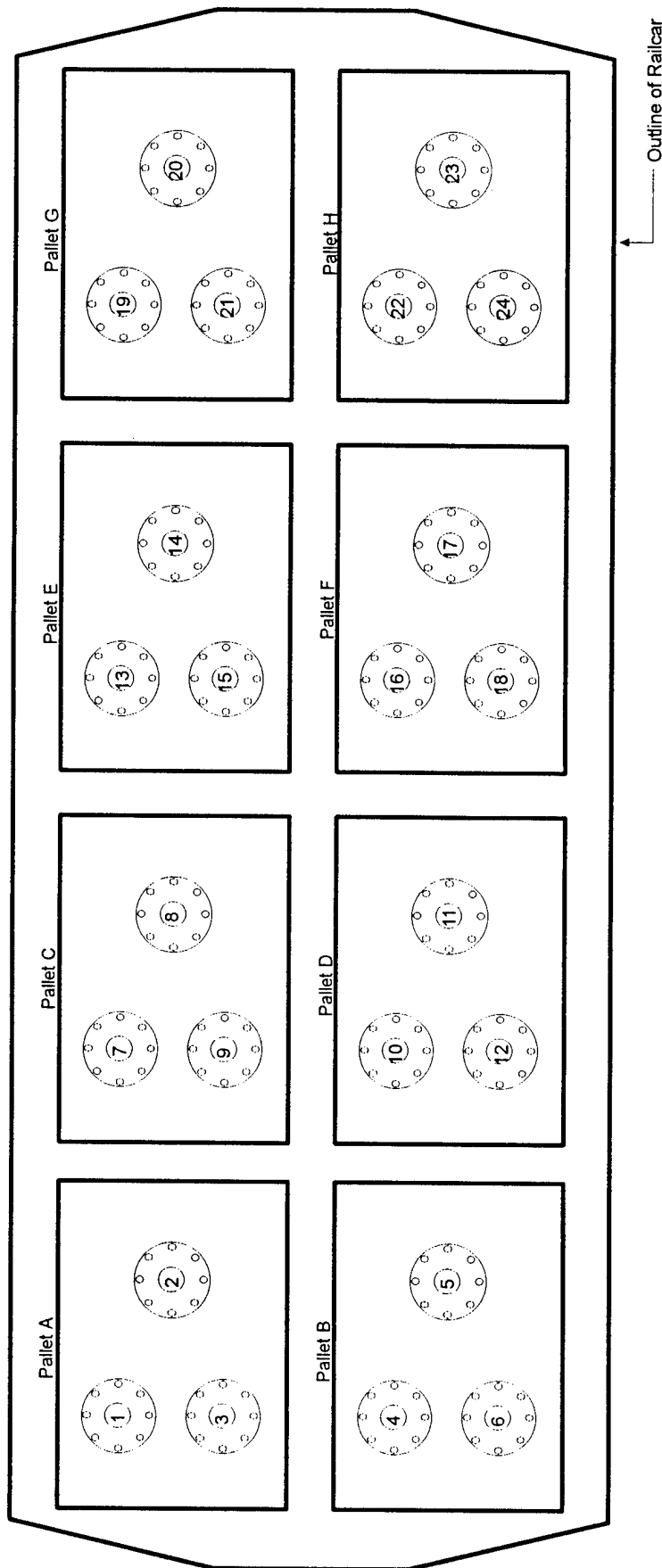
Unit Weight, sawed end = 16 lbs

Total Weight, 24 pieces = 384 lbs

## MK 54 Depth Bombs

(Sawed ends contain HBX residue)

Door Opening



1 Spiked pieces to be sampled

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-7 for sawed ends placement on pallets

Test 16

16 Hours at 750 degrees F.

Figure D-27 MK 54 Depth Bombs (Sawed Ends) with HBX Residue

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

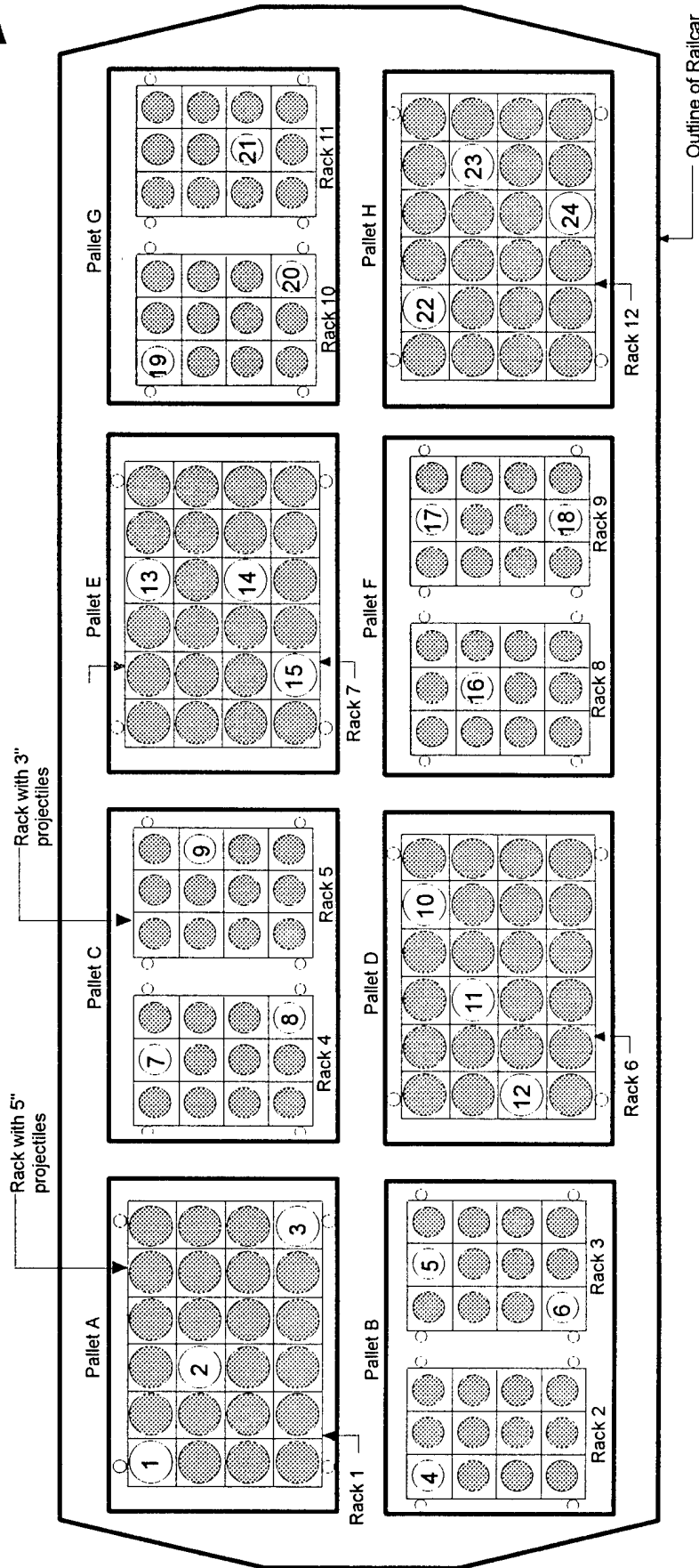
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



1 Spiked projectile to be sampled

● Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-3 for 3" rack placement on pallets  
See Figure D-4 for 5" rack placement on pallets

Test 17

Based on results of Test 12

Figure D-28 3"/5" Projectiles Spiked with RDX

Car Capacity = 11,000 lbs.

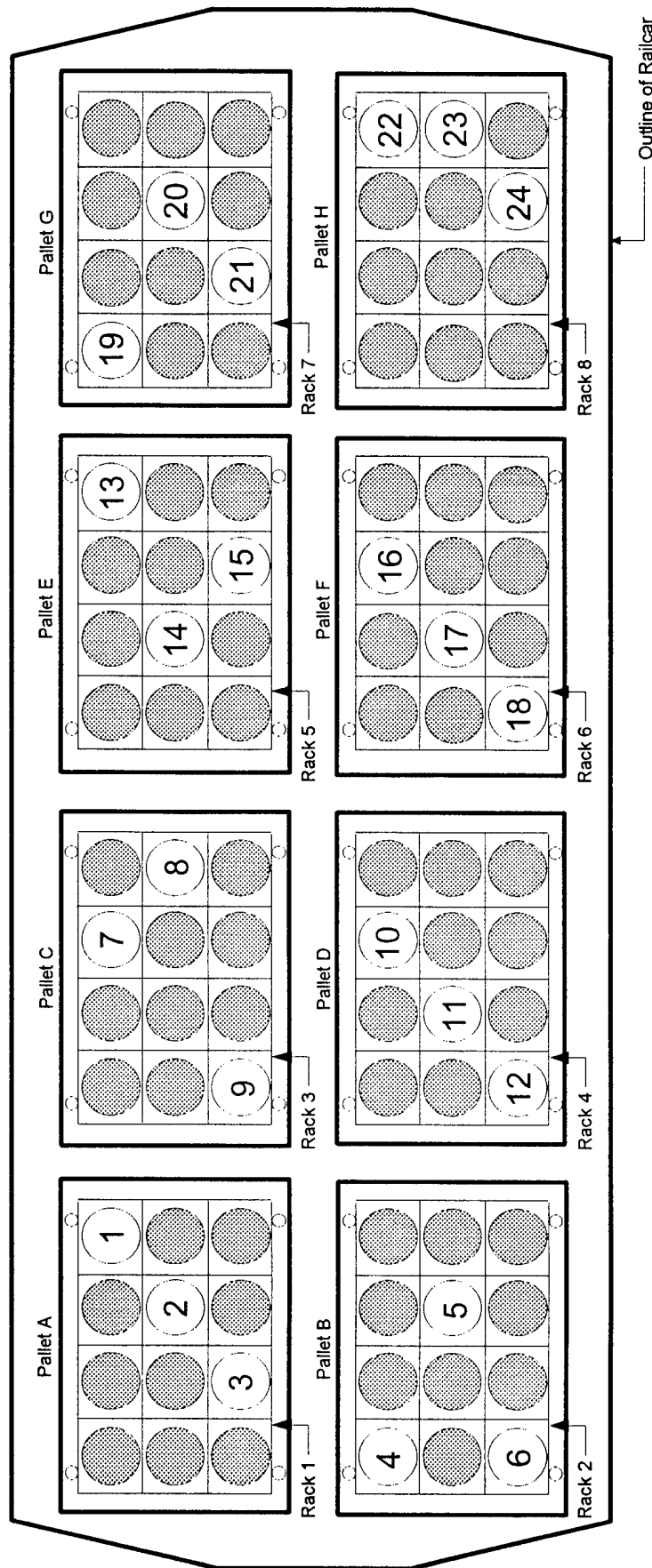
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

## 175mm Projectiles

(Use items from FF-13)

Door Opening



2 Spiked projectile to be sampled

Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test 18

Based on results of Test 13

Figure D-29 175mm Projectiles Spiked with Comp B



Car Capacity = 11,000 lbs.

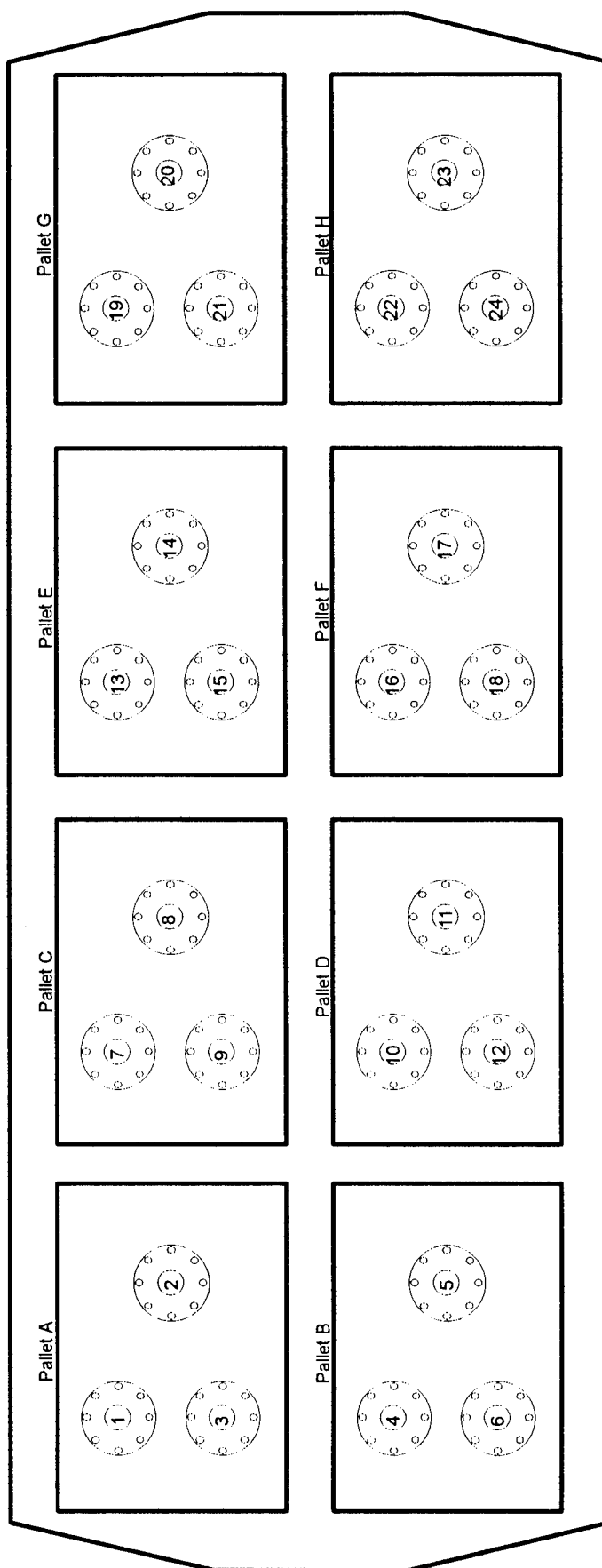
Unit Weight, sawed end = 16 lbs

Total Weight, 24 pieces = 384 lbs

## MK 54 Depth Bombs

(Sawed ends contain HBX residue)

Door Opening



Outline of Railcar

1 Spiked pieces to be sampled

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-7 for sawed ends placement on pallets

Test 19

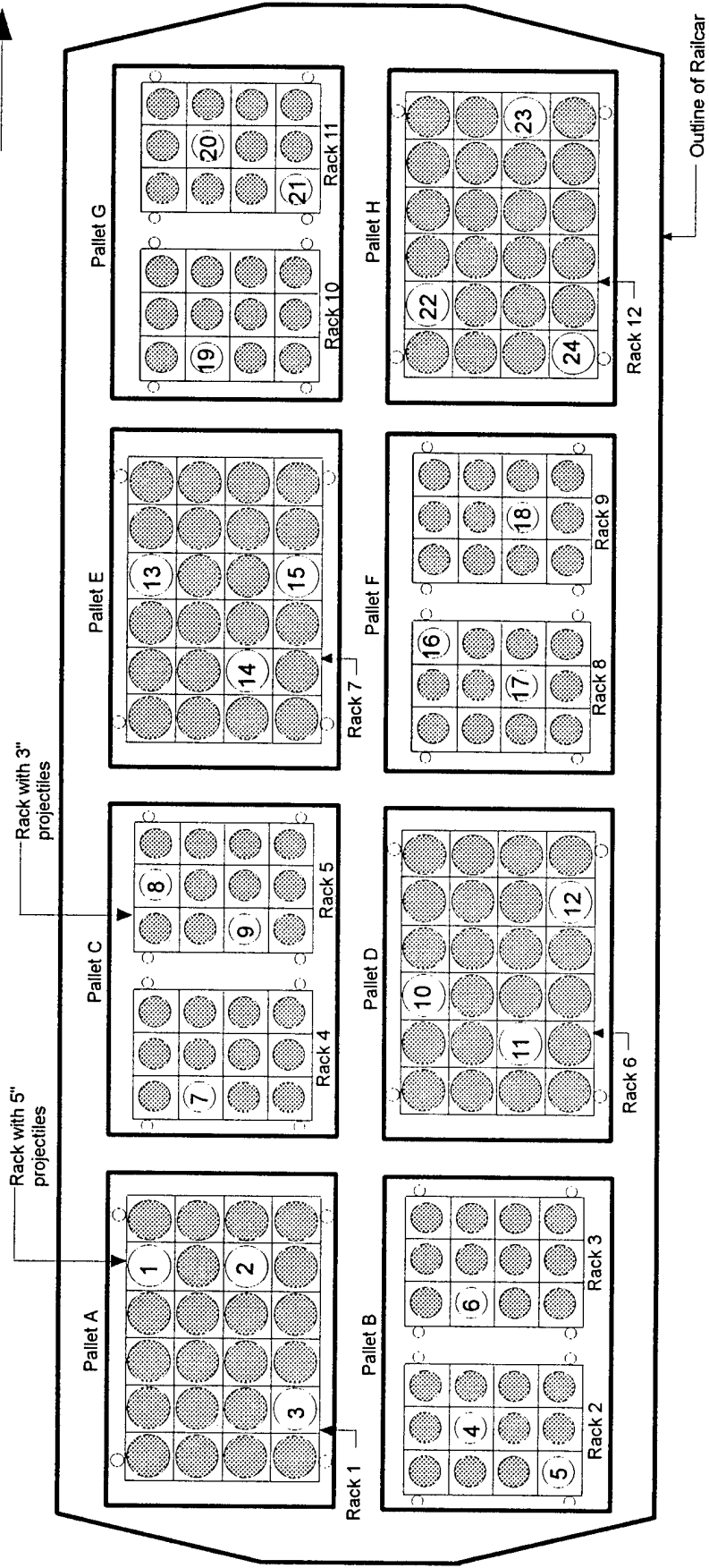
Based on results of Test 16

Figure D-30 MK 54 Depth Bombs (Sawed Ends) with HBX Residue

Car Capacity = 11,000 lbs.  
 Unit Weight, 3" projectile = 9 lbs  
 Unit Weight, 5" projectile = 67 lbs  
 Total Weight, 192 projectiles = 7,296 lbs

# 3"/5" Projectiles (Use items from FF-13)

Door Opening



- 9 Spiked projectile to be sampled
- Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-3 for 3" rack placement on pallets  
 See Figure D-4 for 5" rack placement on pallets

Test 20

Based on results of Test 15

Figure D-31 3"/5" Projectiles Spiked with Yellow D

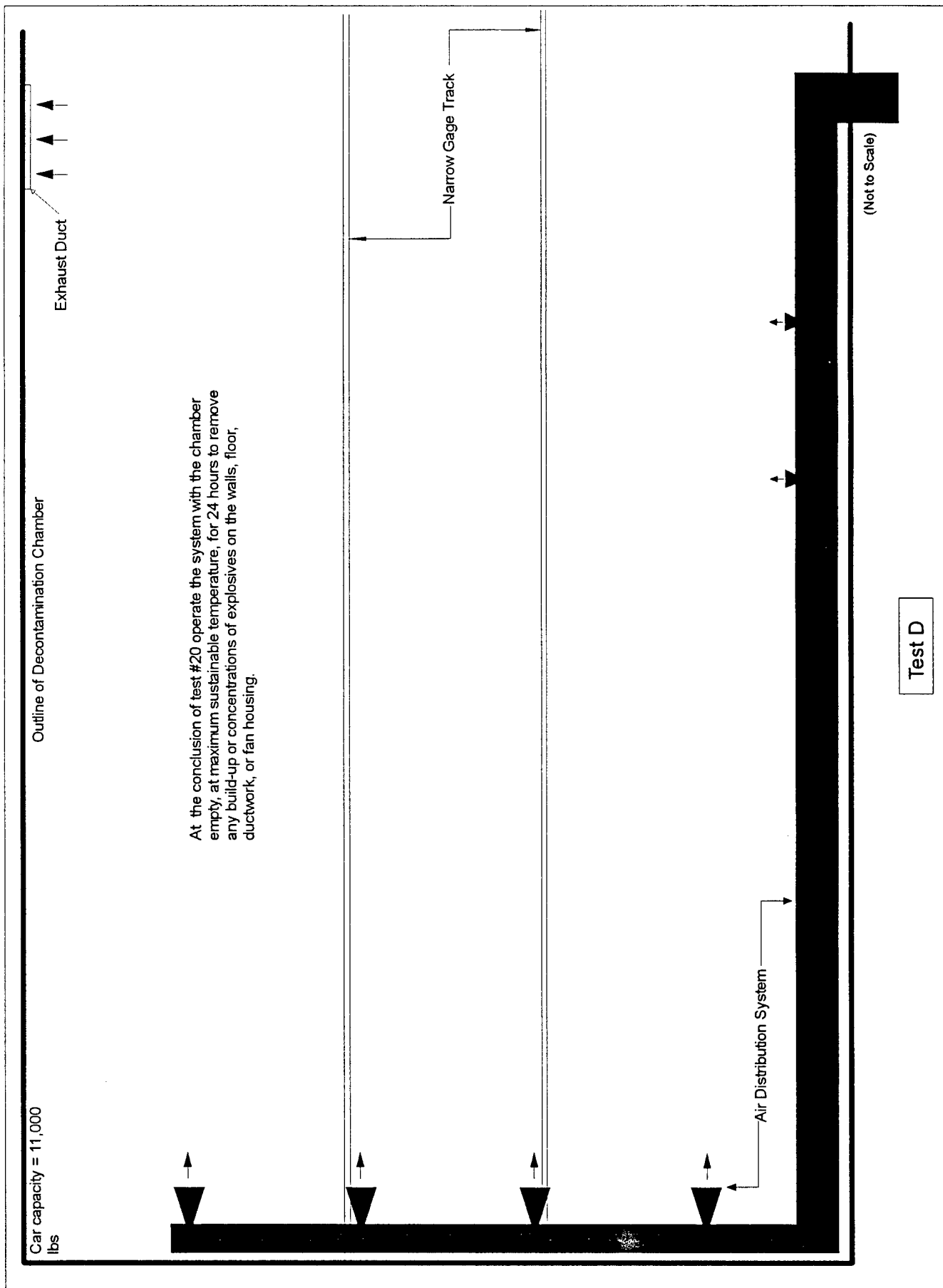


Figure D-32 Empty Chamber Run

Car Capacity = 11,000 lbs.

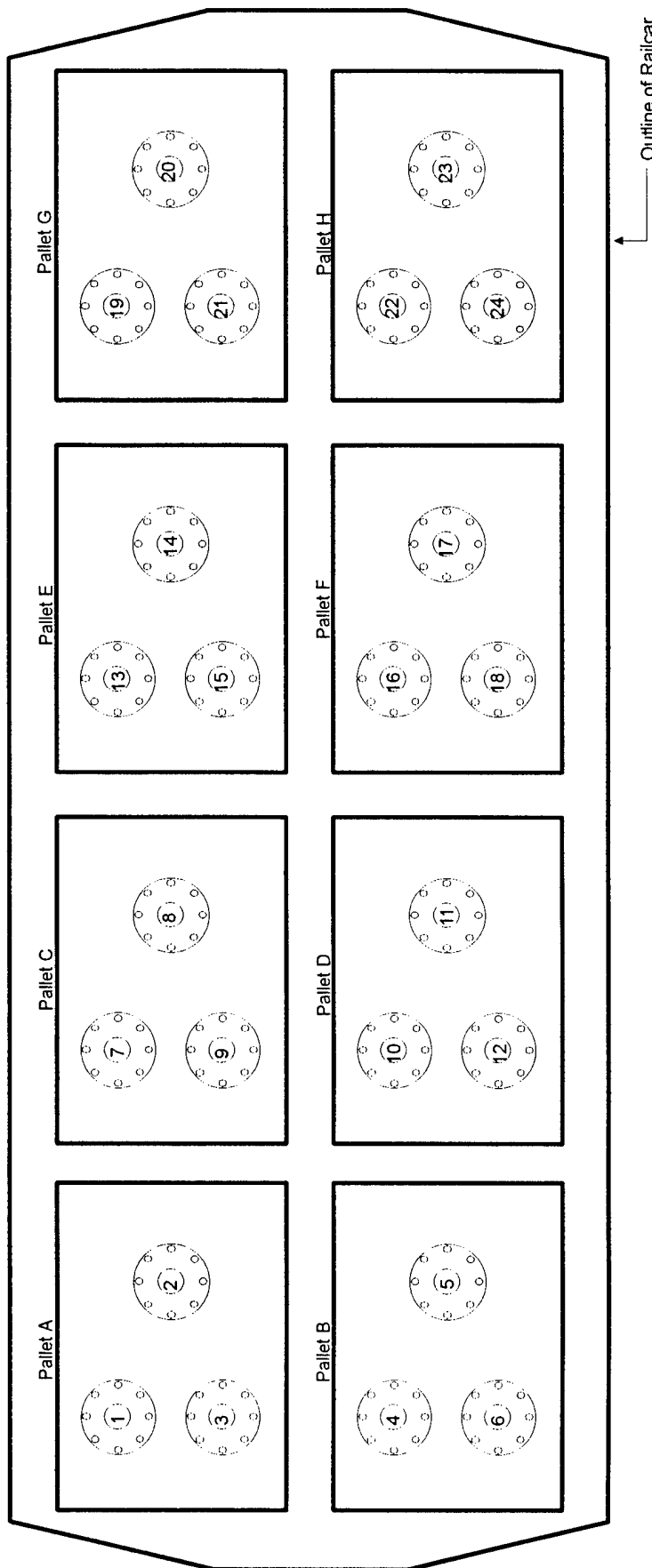
Unit Weight, sawed end = 16 lbs

Total Weight, 24 pieces = 384 lbs

## MK 54 Depth Bombs

(Sawed ends contain HBX residue)

Door Opening



1 Spiked pieces to be sampled

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-7 for sawed ends placement on pallets

Test 21

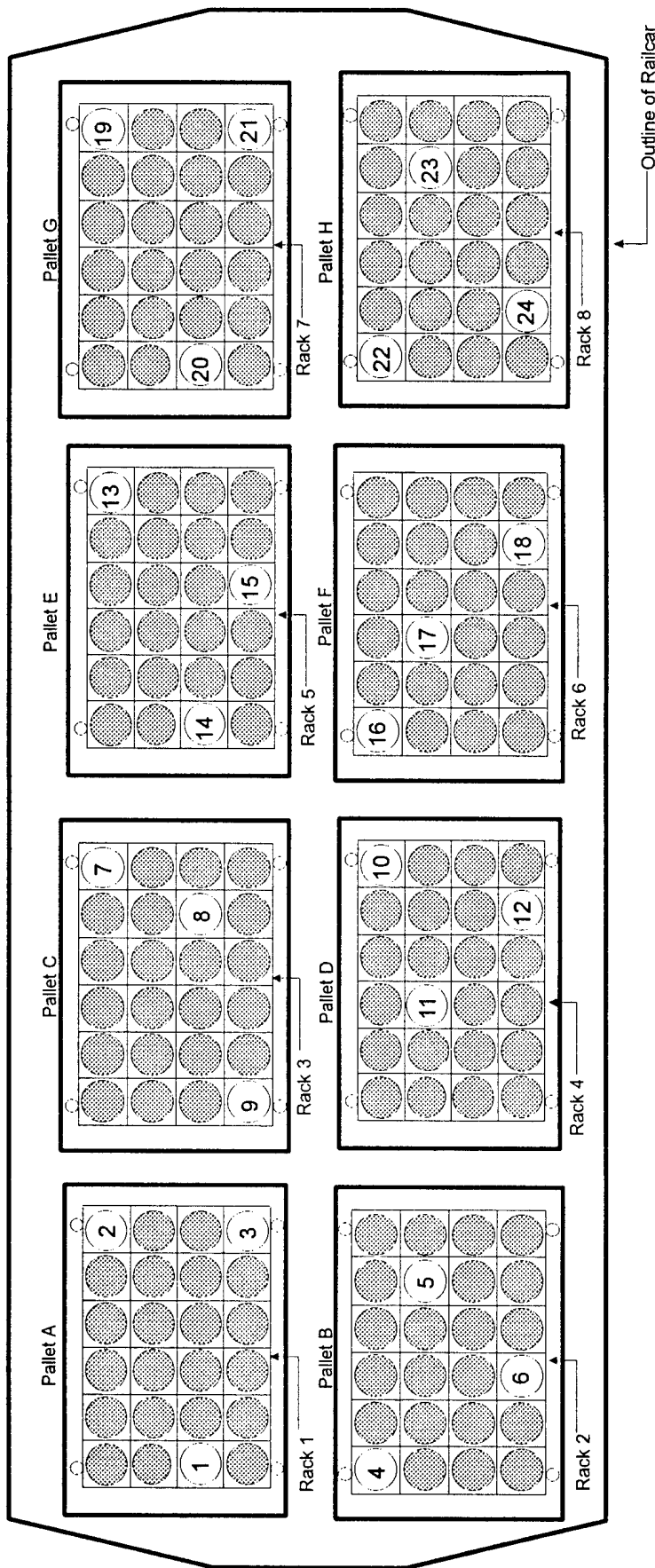
Based on results of Test 19

Figure D-33 MK 54 Depth Bombs (Sawed Ends) with HBX Residue

Car Capacity = 11,000 lbs.  
Unit Weight, 106mm projectile = approx 55 lbs.  
Total Weight, 192 projectiles = 10,560 lbs

# 106mm Projectiles (Projectiles contain Comp A-3 residue)

Door Opening →



- 7 Contaminated projectile to be sampled
- Inert 106mm projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for 5" rack placement on pallets

Test 22

Based on results of Test 17

Figure D-34 106mm Projectiles with Comp A-3 Residue

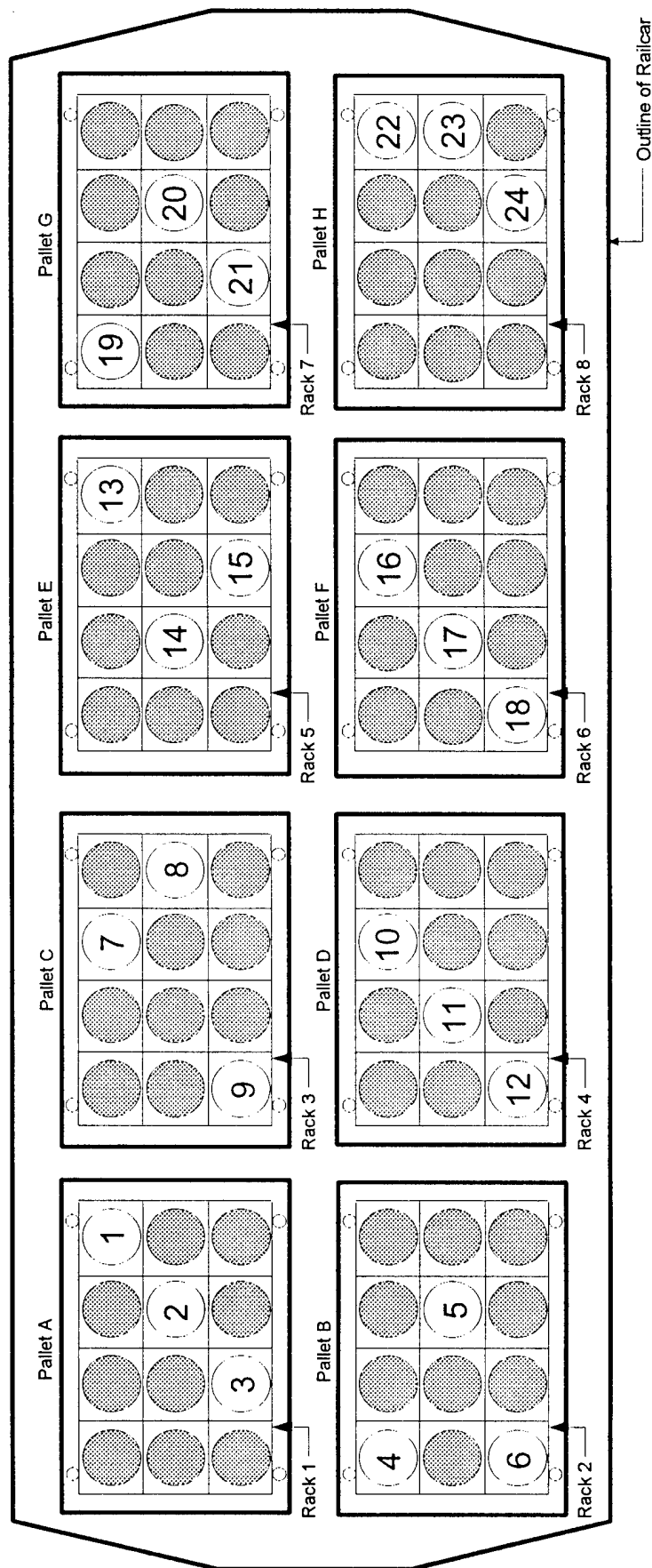
Car Capacity = 11,000 lbs.

Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

# 175mm Projectiles (Projectiles contain Comp B residue)

Door Opening



2 Spiked projectile to be sampled

Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test 23

Based on results of Test 18

Figure D-35 175mm Projectiles with Comp B Residue

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

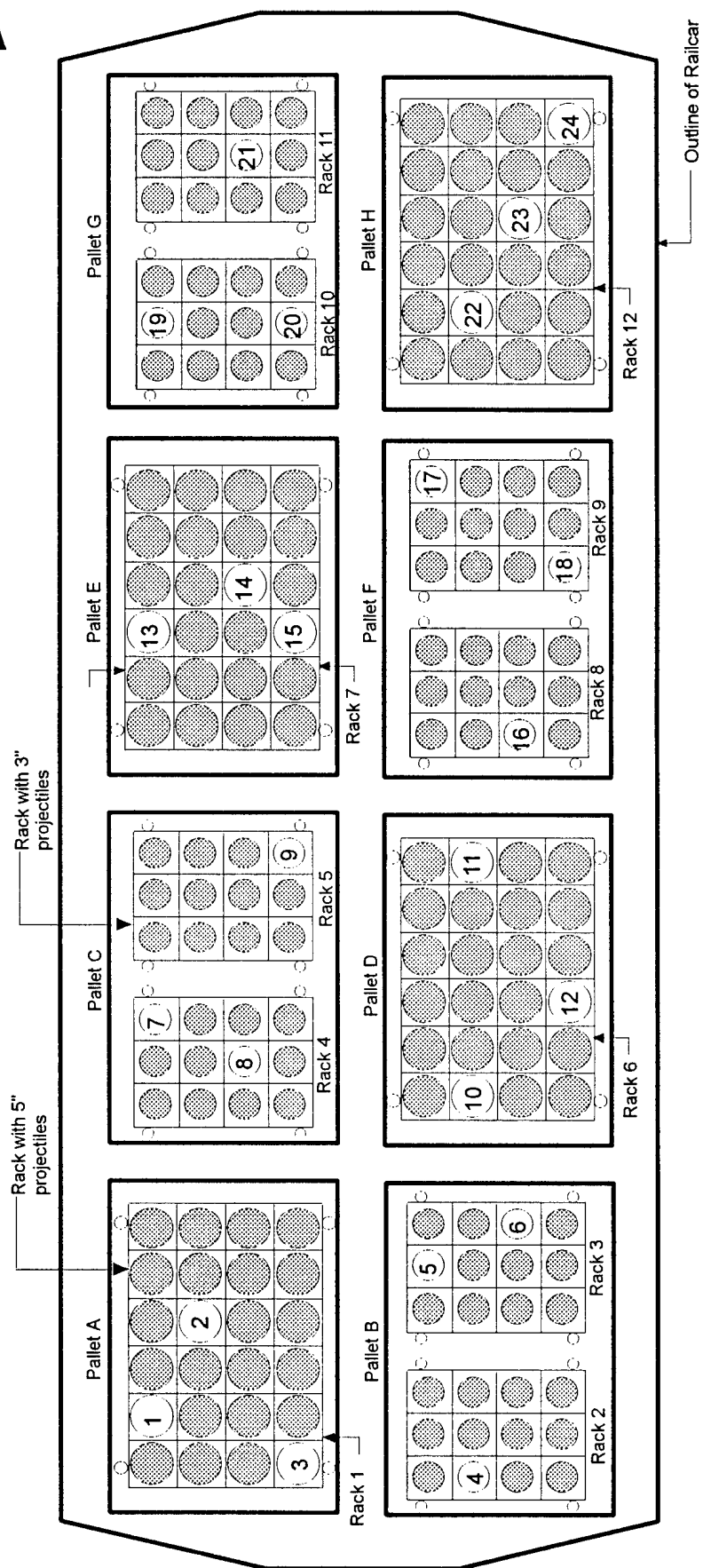
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



2 Spiked projectile to be sampled

Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-3 for 3" rack placement on pallets  
See Figure D-4 for 5" rack placement on pallets

Test 24

Based on results of Test 20

Figure D-36 3"/5" Projectiles Spiked with Yellow D

Car Capacity = 11,000 lbs.

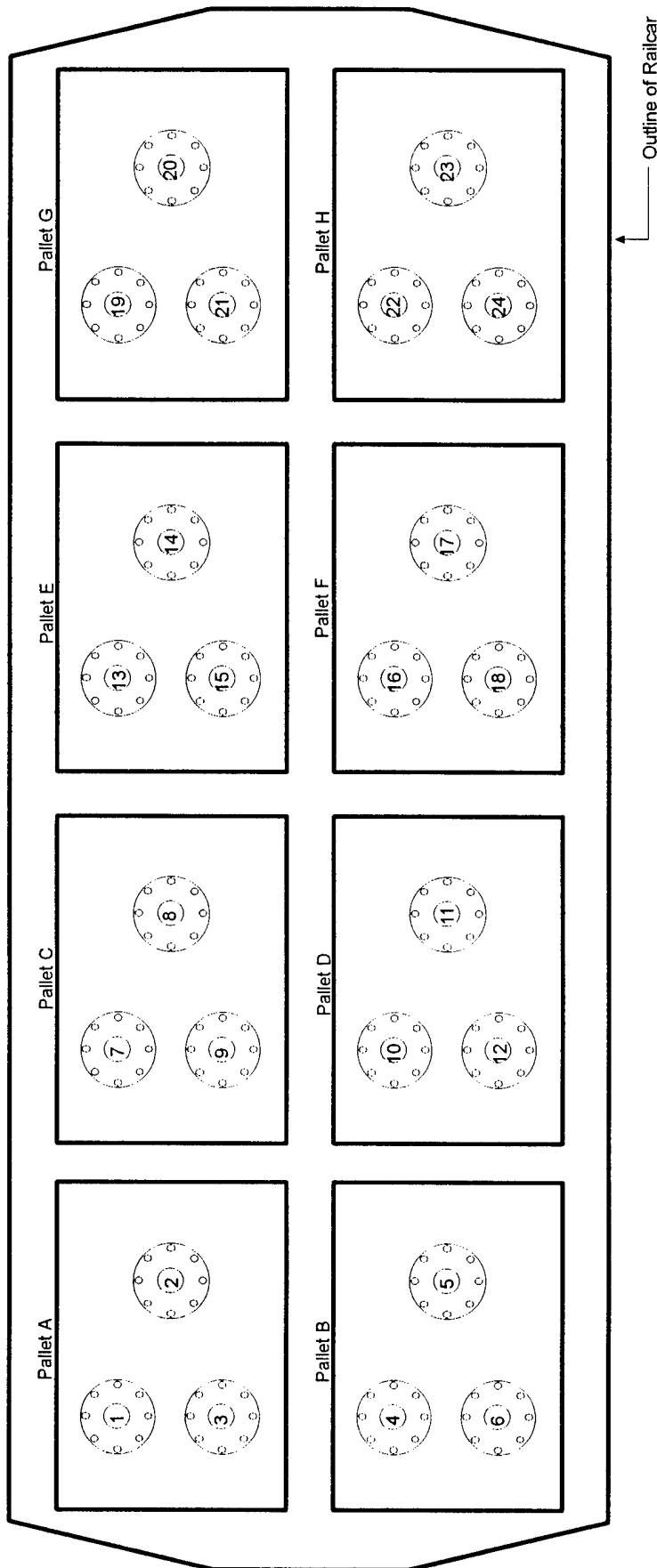
Unit Weight, sawed end = 16 lbs

Total Weight, 24 pieces = 384 lbs

## MK 54 Depth Bombs

(Sawed ends contain HBX residue)

Door Opening



(15) Spiked pieces to be sampled

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-7 for sawed ends placement on pallets

Test 25

Based on results of Test 21

Figure D-37 MK 54 Depth Bombs (Sawed Ends) with HBX Residue



Car Capacity = 11,000 lbs.

Unit Weight, MK 25 = 715 lbs

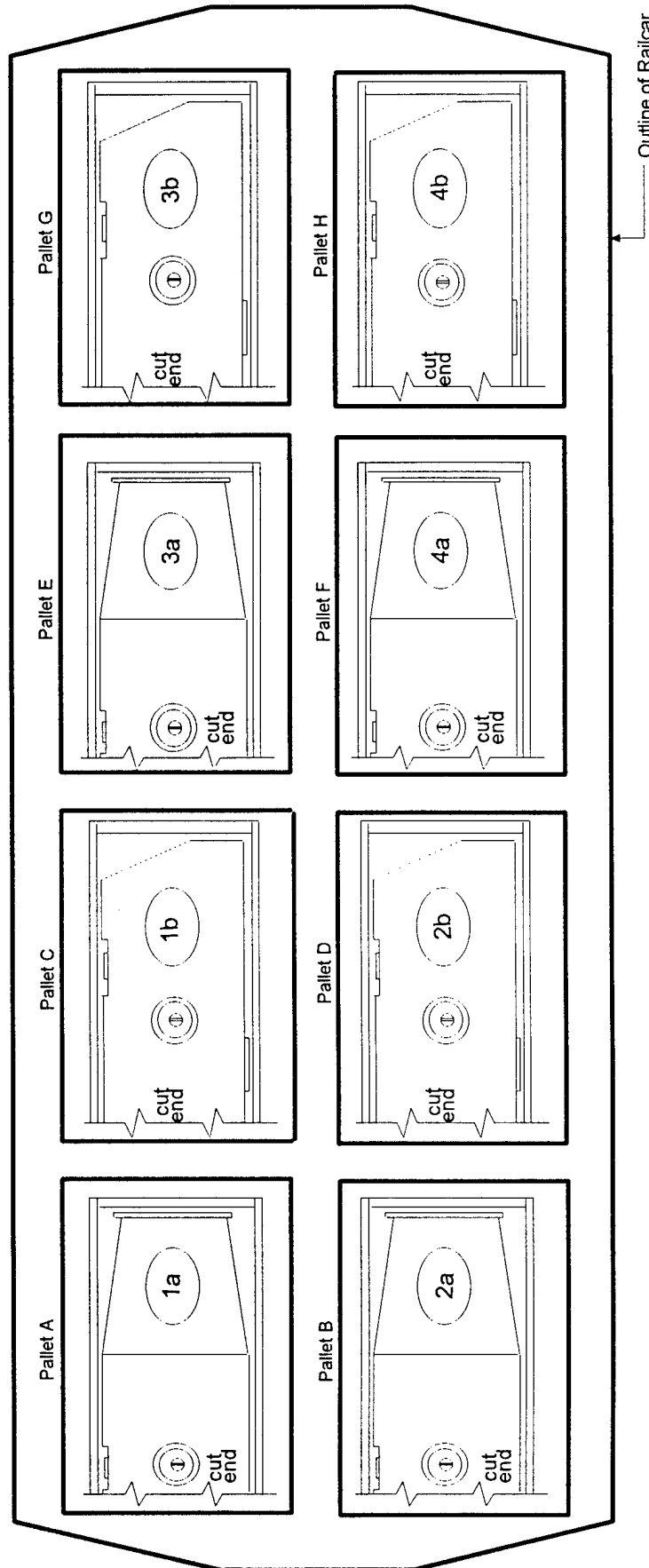
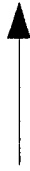
Total Weight, 4 Mines = 2,860 lbs

## MK 25 Ship Mines

(Unused mines - internals coated with hot-melt and spiked with TNT)

[Left over from previous testing]

Door Opening



4b Spiked mine half to be sampled

Test 26

Based on results of Test 11

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-8 for mine placement on pallets

Figure D-38 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

Car Capacity = 11,000 lbs.

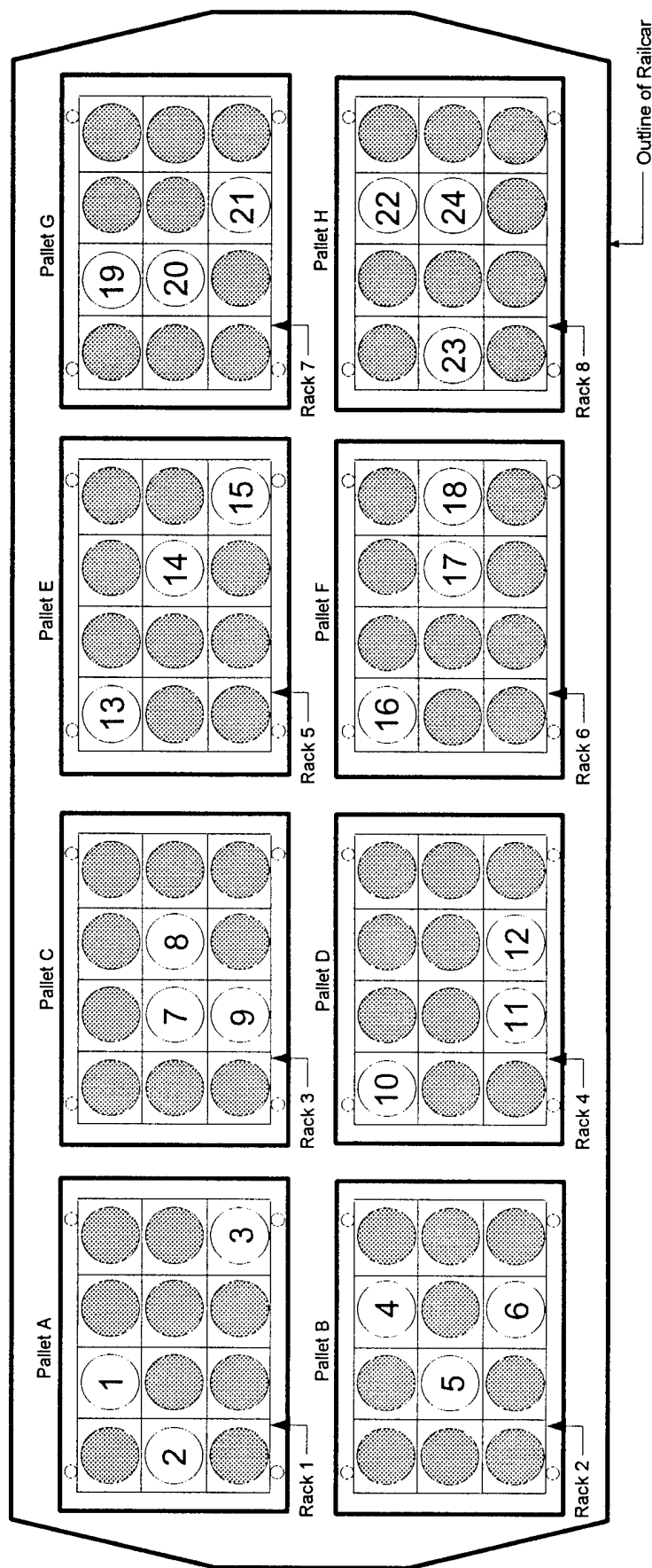
Unit Weight, 175mm projectile = 115 lbs

Total Weight, 96 Projectiles = 11,040 lbs

# 175mm Projectiles

(Projectiles containing Comp B residue)

Door Opening



15 Spiked projectile to be sampled

Inert projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-5 for rack placement on pallets

Test 27

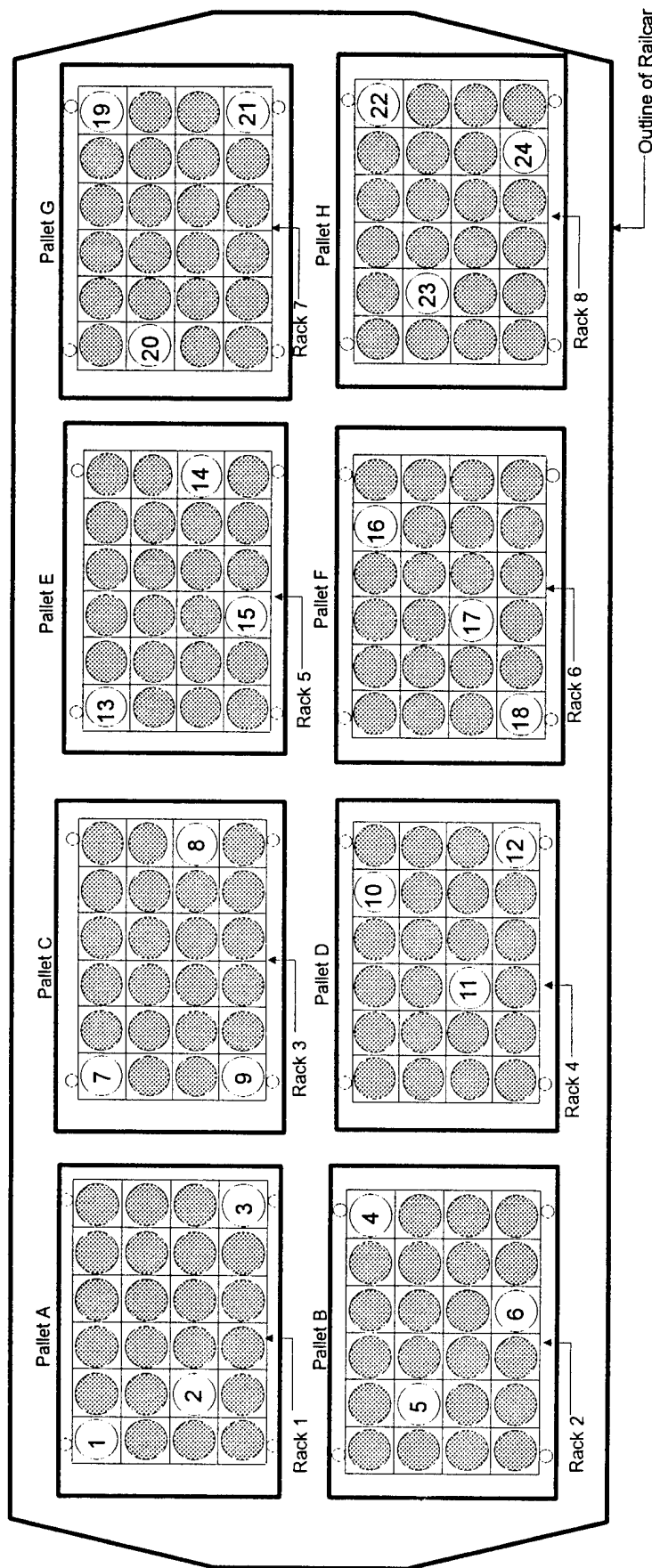
Based on results of Test 23

Figure D-39 175mm Projectiles with Comp B Residue

Car Capacity = 11,000 lbs  
 Unit Weight, 106mm projectile = approx 55 lbs.  
 Total Weight, 192 projectiles = 10,560 lbs.

# 106mm Projectiles (Projectiles contain Comp A-3 Residue)

Door Opening



- 7 Contaminated projectile to be sampled
- Inert 106mm projectile added for thermal mass

See Figure D-1 for railcar placement in chamber  
 See Figure D-2 for pallet placement on railcar  
 See Figure D-5 for 5' rack placement on pallets

Test 28

Based on results of Test 22

Figure D-40 106mm Projectiles with Comp A-3 Residue

Car Capacity = 11,000 lbs.

Unit Weight, 3" projectile = 9 lbs

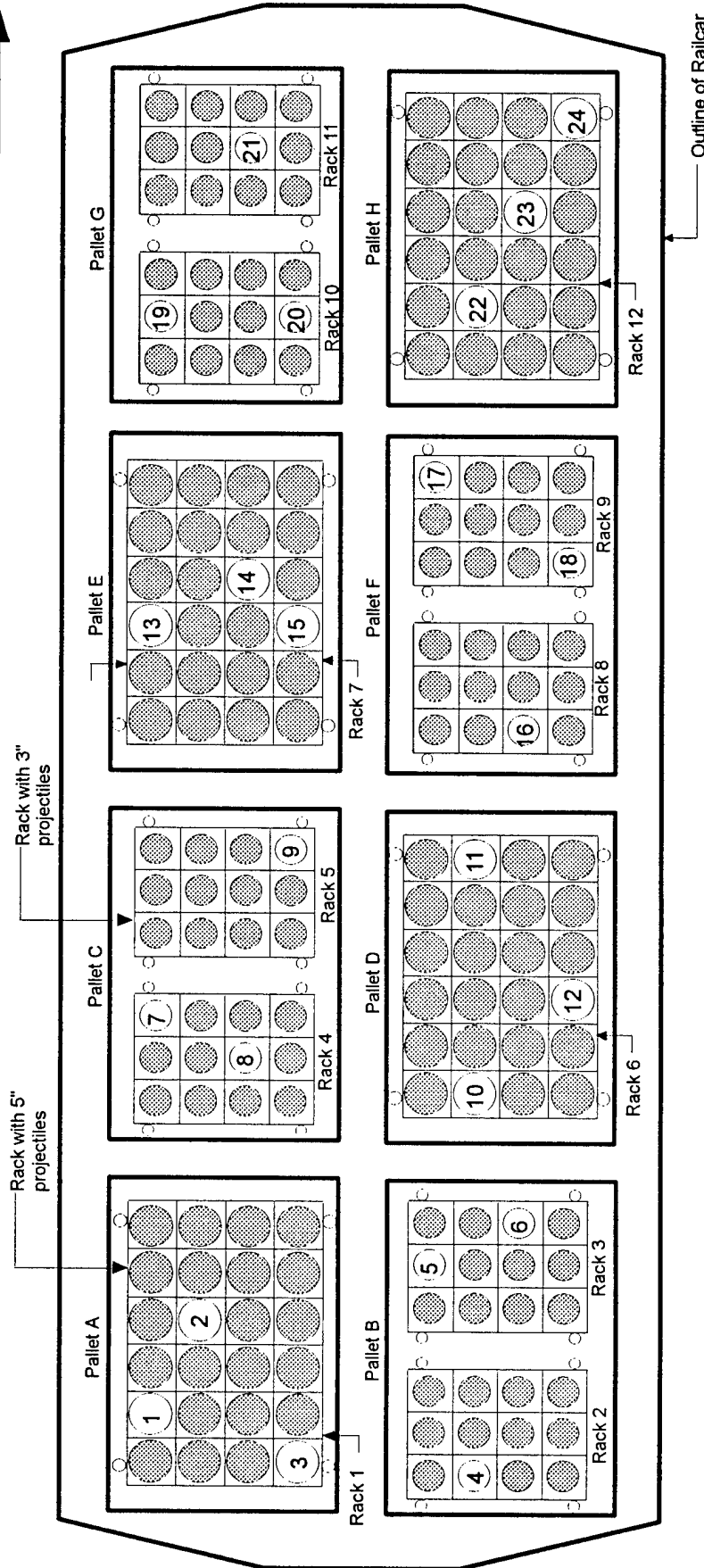
Unit Weight, 5" projectile = 67 lbs

Total Weight, 192 projectiles = 7,296 lbs

## 3"/5" Projectiles

(Use items from FF-13)

Door Opening



Test 29

Based on results of Test 24

See Figure D-1 for railcar placement in chamber

See Figure D-2 for pallet placement on railcar

See Figure D-3 for 3" rack placement on pallets

See Figure D-4 for 5" rack placement on pallets

Figure D-41 3"/5" Projectiles Spiked with Yellow D

Car Capacity = 11,000 lbs.

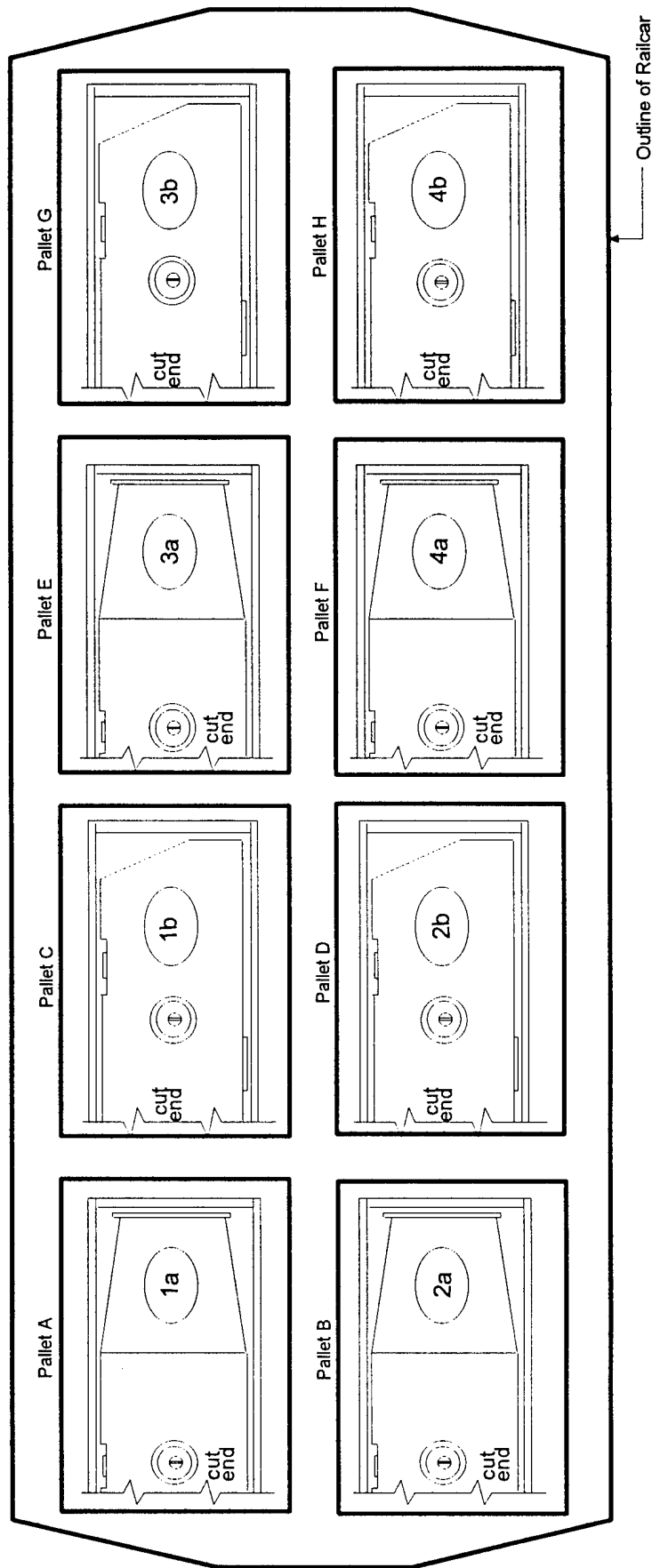
Unit Weight, MK 25 = 715 lbs

Total Weight, 4 Mines = 2,860 lbs

## MK 25 Ship Mines

(Unused mines - internals coated with hot-melt and spiked with TNT)  
[Left over from previous testing]

Door Opening



3a Spiked mine half to be sampled

See Figure D-1 for railcar placement in chamber  
See Figure D-2 for pallet placement on railcar  
See Figure D-8 for mine placement on pallets

Test 30

Based on results of Test 26

Figure D-42 MK 25 Ship Mines Hot-Melt Coated Internals and Spiked with TNT

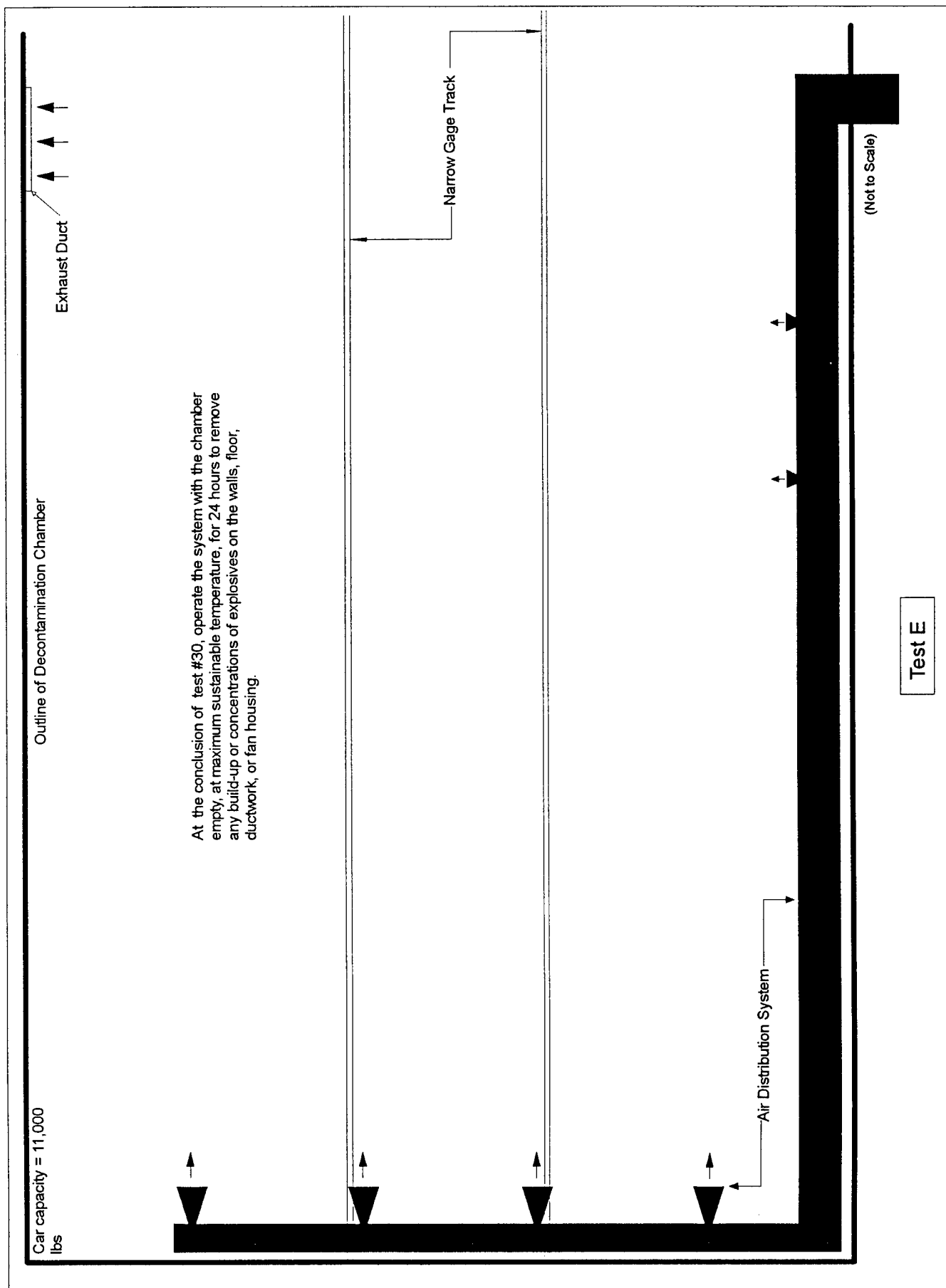


Figure D-43 Empty Chamber Run

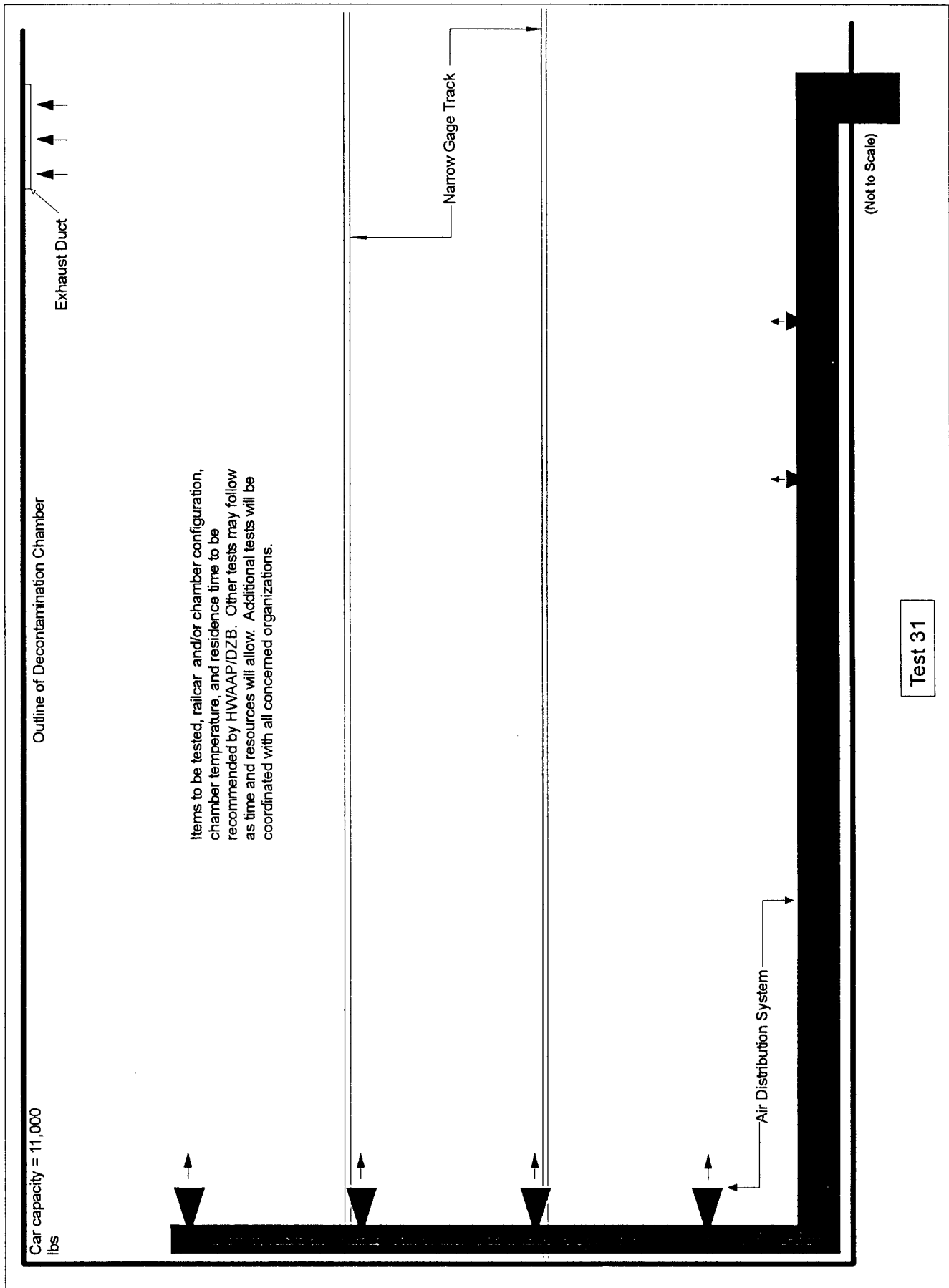


Figure D-44 Test to be Determined by HWAAP/DZB

## **APPENDIX E**

### **LABORATORY CONTROL DOCUMENTS**



## LABORATORY CONTROL DOCUMENTS

- E1 Procedure HGD-0001 - "Spiking Explosives on Metal Surfaces"
- E2 Procedure HGD-0002 - "Extraction Experiments"
- E3 Procedure HGD-0003 - "Planning Sampling Activities and Sampling"
- E4 Procedure HGD-0004 - "Safety and Emergency Plans"
- E5 Procedure HGD-0005 - "Use of Explosives Storage Room"
- E6 Procedure HGD-0006 - "Method Detection Limits"

This appendix includes the current versions of TVA laboratory control documents describing sampling and analytical activities. These documents will be updated and revised as methods development and process testing progress. The definitive updated versions will be maintained on file at TVA Muscle Shoals and revisions of applicable sections will be forwarded to the TVA field team at HWAAP. At the conclusion of the HGD test and demonstration, the latest versions of these documents will be included in the HGD final report, together with a discussion of the significant revisions made during the program.

**APPENDIX E1**

**PROCEDURE HGD-0001**

**SPIKING EXPLOSIVES ON METAL SURFACES**

## **SPIKING EXPLOSIVES ON METAL SURFACES**

### **1.0 PURPOSE**

This procedure describes those actions required to uniformly spike standard material on a metal surface or shell to simulate uniform deposition of contaminants left after steam cleaning of the metal surface.

### **2.0 SCOPE**

This procedure applies to work done in support of the Hot Gas Decontamination Project.

It is followed by an extraction or smear procedure to test removal of the spiked compound for quantitative recovery. Alternately, it may be followed by other experimental activities such as heat treatment to remove spiked compounds.

### **3.0 SUMMARY**

Three methods of spiking are described:

An explosive material is dissolved and allowed to run down the side of the shell.

An explosive material is placed in a shell with suitable solvent to make a solution.

An explosive material is dissolved and then deposited uniformly in a scribed area.

In each case, the solvent is allowed to evaporate, leaving a film of the material behind.

**4.0      REFERENCES**

- 4.1**      "Dangerous Properties of Industrial Materials," Seventh Edition, N. Irving Sax and Richard J. Lewis, Sr., Van Nostrand Reinhold, New York.

**5.0      RESPONSIBILITIES**

It is the responsibility of the supervisor to ensure this procedure is followed and that employees utilize appropriate safety precautions.

It is the responsibility of the technician to follow this procedure, including the safety precautions. It is the responsibility of the technician to record all data and to report unusual results to the supervisor.

**6.0      PROCEDURE/REQUIREMENTS**

**6.1      Prerequisites**

Employees handling the quantities of explosive utilized in this procedure shall have had prior safety training in the handling of explosive materials.

**6.2      Limitations and Actions**

- 6.2.1**      The "Spike and Rinse" procedure steps are utilized when an extremely large item is spiked which cannot be rotated easily.

- 6.2.2**      The "*In-Situ* Dissolution" procedure steps are used for moderate sized items which can be rotated or agitated. The purpose of this section is to eliminate the need for multiple rinses of a weighing vessel and delivery device to ensure all the material is transferred.

- 6.2.3**      The "Spiking a Scribed Area" procedure steps are used for items which are very large and cannot be moved at all and which will have access by a small port. It may also be used to spike small plates of metal which are tested by various extraction, washing, or heating processes for removal of material.

- 6.2.4**      Weighings of target compounds should be made to 0.0001 g for total weights of less than 1 gram, to 0.001 g for total weights of less than 10 grams, and to 0.01 g for larger weights.

### **6.3        Requirements**

#### **6.3.1     Apparatus/Equipment**

1-liter flask with ground glass stopper

1-liter Erlenmeyer flask

50-milliliter volumetric pipett

Analytical balance, calibrated, capable of weighing to 0.0001 g.

Laboratory balance, calibrated, capable of weighing to 0.001 g.

Technical balance, calibrated, capable of weighing to 0.01 g.

Gloves impermeable to the solvents.

Teflon plug machined to fit the shell fuze opening.

Non-sparking

Area template (10 cm by 10 cm) or other size used to scribe an area to be spiked.

Glass dropper

#### **6.3.2     Reagents and Standards**

**6.3.2.1    Acetone, HPLC grade**

**6.3.2.2    Acetonitrile, HPLC grade**

**6.3.2.4    Methanol, HPLC grade**

**6.3.2.5    Reagent grade water**

**6.3.2.6    Target compound (an explosive compound to be studied as provided by USAEC)**

**6.4**      **Calibration**

The balances should be calibrated with traceability to the National Institute of Standards and Technology.

**6.5**      **Procedure Instructions**

CAUTION: Wear suitable gloves while performing the following steps. Avoid inhaling fumes. Work in an area with adequate ventilation. When spiking small components, work in a fume hood.

CAUTION: Wear eye protection to prevent splashes from getting in the eye.

CAUTION: The solvents are flammable and very volatile. Do not use near sources of heat, sparks, or ignition. Keep containers tightly closed when not in use.

CAUTION: In case of skin exposure, wash immediately with soap and water.

**6.5.1**      **Spike and Rinse Process**

**6.5.1.1**      Weigh a prescribed quantity (initially 10 grams) of target compound into a 1-liter flask. Record the weight.

**6.5.1.2**      Add the solvent ( methanol for Yellow-D, acetonitrile for all others) to fill approximately 90 percent of the volume of the flask. Stopper the flask and shake to dissolve. Invert periodically while shaking to ensure complete mixing.

**6.5.1.3**      Incline the shell to be spiked so that it is nearly horizontal.

**6.5.1.4**      Fill the pipette and allow the solution to drain down the side of the shell. If solution reaches the bottom of the object, reduce the angle of inclination so that it runs back on the side.

6.5.1.5 Allow the solvent to evaporate and form a thin film of the target compound on the side of the shell.

6.5.1.6 Rotate the shell so that a fresh surface is exposed and pipette more solution into it.

6.5.1.7 Repeat this until all the solution is used.

6.5.1.8 Rinse the flask with approximately 50 ml of solvent and pipette it onto the surface in a similar manner.

6.5.1.9 Repeat 6.5.1.8 with two more 50 ml portions of solvent.

6.5.2 *In-situ* Dissolution

6.5.2.1 Weigh a container plus the prescribed amount (initially 10g) of the target compound. Record the weight.

6.5.2.2 Pour the target compound into the shell and weigh the empty container. Record the weight.

6.5.2.3 Add a suitable portion of solvent (200 to 500 ml) to the shell to dissolve the compound. Stopper the shell fuze opening with a teflon plug of appropriate size. Agitate the shell slightly to enhance dissolution.

6.5.2.4 Remove the teflon plug and rinse the surface exposed to the solution into the shell with two or three small portions of solvent.

6.5.2.5 Incline the shell to nearly horizontal and slowly rotate the shell to allow a thin film of solution to flow along the shell wall. Allow the solvent to evaporate while the shell is rotating to form a thin film of target compound on the wall of the shell.

6.5.3 Spiking a Scribed Area

- 6.5.3.1 Scribe a suitable area for testing ( usually 100 square centimeters) with the metal scribe. Utilize a template if one is available.
- 6.5.3.2 Weigh the target compound into the flask to the nearest 0.0001 g.
- 6.5.3.3 Add the smallest amount of solvent required to dissolve the compound.
- 6.5.3.4 Using a dropper, drop the solution into the scribed area and allow it to evaporate. Ensure the entire area is covered, but do not allow the solution to run outside the scribed area.
- 6.5.3.5 When all the solution is placed on the scribed area, rinse the flask and dropper three times with a small amount of solvent, dropping the rinsate into the scribed area as above.

## 6.6 Calculating and Reporting Data

Report weights utilized in spiking. When weights are calculated by difference, report initial and final weights and the difference.

## 7.0 QUALITY ASSURANCE PROVISIONS

### 7.1 Responsibility of Inspection

### 7.2 Acceptance Criteria

The dried material should be uniformly spread over the surface to be studied without visible bare patches, lumps, or accretions.

### 7.3 Material Monitoring

None

### 7.4 Equipment Monitoring

None

### 7.5 Certification

This procedure is certified by the review and approval process.



**7.6**      **Quality Control Sample Requirements**

None

**8.0**      **SAFETY**

**8.1**      Solvents utilized in this procedure have some hazards associated with them as summarized below. Also reference any Material Safety Data Sheets present in the work area.

**8.1.4**      Acetone is a flammable liquid. It is moderately toxic by various routes. It is a skin irritant and severe eye irritant. OSHA Permissible Exposure Limit is 1000 ppm. The Short Term Exposure Limit is 1000 ppm. It can react vigorously with oxidizing materials. It is commercially used as nail polish remover where its defatting action on skin is familiar.

**8.1.2**      Acetonitrile is a flammable liquid which is poisonous by ingestion and intraperitoneal routes. It is a skin and severe eye irritant. It is a dangerous fire hazard when exposed to heat, flame, or oxidizers. It reacts with water. OSHA Permissible Exposure Limit is 40 ppm (70 mg/m<sup>3</sup>). The Short Term Exposure Limit is 60 ppm.

**8.1.3**      Methanol is a flammable liquid which is poisonous by ingestion. Some experimental data show that it is poisonous by skin contact. It is mildly toxic by inhalation. Both by ingestion and inhalation it may attack the optic nerve. It is an eye and skin irritant. Death from ingestion of 30 ml has been reported. It is a dangerous fire hazard when exposed to heat, flame, or oxidizers.

**8.1.4**      They should be utilized in a laboratory hood for small objects and in a well ventilated area for large objects. A general rule of thumb is that if an odor is detected, the TLV has been violated. If an odor is detected, wear a respirator.

**8.1.5**      In case of skin contact, wash immediately with soap and water.

**8.1.6** In case of eye contact, flush immediately with water, holding the eyes open to ensure they are rinsed.

**8.2** Gloves impenetrable to the particular solvent must be worn when handling it.

**8.3** The quantities of explosives utilized in this procedure are large enough quantities to constitute a hazard. No flames, sparks, or sparking materials shall be allowed in their presence. When working with the dry powders, electrical grounding shall be utilized to prevent sparks from static electricity. Avoid excess friction or impact.

**9.0** NOTES  
None

**10.0** ATTACHMENTS AND APPENDICES

**APPENDIX E2**

**PROCEDURE HGD-0002**

**"EXTRACTION EXPERIMENTS"**

Procedure HGD-0002 Extraction Experiments

**1.0**      **PURPOSE**

This procedure is intended to describe actions used to investigate and develop an extraction procedure to remove explosive materials from a spiked metal surface.

**2.0**      **SCOPE**

This procedure applies to the investigative phase of the Hot Gas Decontamination Project.

**3.0**      **SUMMARY**

A solvent or solvent system is investigated as to its effectiveness in removing various target compounds from metal surfaces both as a function of composition and a function of time.

Smear effectiveness is also investigated.

**4.0**      **REFERENCES**

**4.1**      "Dangerous Properties of Industrial Materials," Seventh Edition, N. Irving Sax and Richard J. Lewis, Sr., Van Nostrand Reinhold, New York

**4.2**      "Safety and Emergency Plan", HGD-0004, Analytical Laboratory Support Services, Tennessee Valley Authority

**5.0**      **RESPONSIBILITIES**

It is the responsibility of the supervisor to ensure this procedure is followed and that employees utilize appropriate safety precautions.

It is the responsibility of the technician to follow this procedure, including the safety precautions. It is the responsibility of the technician to record all data and to report unusual results to the supervisor.

**6.0**      **PROCEDURE/REQUIREMENTS**

**6.1**      **Prerequisites**

Spiking is done in accordance with HGD-0001 to place a uniform amount of target compound in the munition or on the metal surface prior to performance of this procedure.

**6.2 Limitations and Actions**

In one portion of the experiments described below, extractions shall be made in sequence until the last extract does not contain the target compound above the detection limit of the measurement process.

**6.3 Requirements**

**6.3.1 Apparatus/Equipment**

Graduated cylinder - laboratory grade

Test logbook - a bound record book utilized for logging experimental data.

Sampling pipette - a glass volumetric pipette.

**6.3.2 Reagents and Standards**

**6.3.2.1 Acetone, HPLC grade**

**6.3.2.2 Acetonitrile, HPLC grade**

**6.3.2.4 Methanol, HPLC grade**

**6.3.2.5 Reagent grade water**

**6.3.2.6 Target compound (an explosive compound to be studied as provided by USAEC)**

**6.4 Calibration**

None

**6.5 Procedure Instructions**

**6.5.1 Extraction as a Function of Time**

Plan the test times and solvent mixture. Devise a log page in the test logbook so that all pertinent data may be easily recorded.

Add a measured volume of extraction solvent or solvent mixture to the munition. Plug with a teflon plug. Record the solvent, quantity, and time.

Agitate by rotating the munition or by shaking in a reciprocating shaker (if the test item is small enough) for the first test period.

Stop the shaker at the first time period and take a 1-ml sample. Place it in a sample vial. Label the vial. Replace the plug and continue shaking.

**Note:** Smaller or larger quantities of sample may be taken as required by the analytical technique. Some samples may be directly sampled in a syringe and injected in the analytical device.

Repeat this for all the pre-planned test points.

Submit the samples for analysis.

Plot the concentration as a function of time.

#### **6.5.1 Extraction as a function of the number of washes**

Plan the number of extractions. Plan the solvent or solvent mixture to be used. Devise a suitable log page in the test log book so that all pertinent data may be easily recorded.

Add a measured volume of extraction solvent or solvent mixture to the munition. Plug with a teflon plug. Record the solvent, quantity, and time.

Agitate by rotating the munition or by shaking in a reciprocating shaker (if the test item is small enough) for the test period.

Stop the shaker at the end of the time and drain the sample into a sample container. Label the container. Add the next volume of solvent. Replace the plug and continue shaking.

Repeat this for the pre-planned number of extractions.

Submit the samples for analysis.

Plot the concentration as a function of the number of extractions.

### 6.5.3 Smear Efficiency

Plan the solvent or solvent mixture and number of smears. Devise a page in the test log book for recording data.

Smear a spiked area with a single smear moistened with a suitable solvent.

Place the smear in a labeled container.

Repeat for the proper number of smears.

Log all actions, times, solvents, and pertinent information.

Submit the samples for analysis.

Plot micrograms (or milligrams) recovered as a function of number of smears.

### 6.6 Calculating and Reporting Data

Mass balance for a series of extractions shall be calculated as follows:

Mass Balance = Mass out / Mass in

where "Mass in" is the mass in milligrams spiked onto a clean surface and "Mass out" is defined as

Mass out =  $C_1 \cdot V_1 + C_2 \cdot V_2 + C_3 \cdot V_3 \dots$

Here  $C_1$  is the concentration from the first extraction in micrograms per liter.  $V_1$  is the volume in the first extraction in L and so forth.

The uncertainty in the mass balance is equal to the square root of the sum of similar terms  $T_i$  as in the following equation:

$$T_i = (C_i \cdot V_i)^2 [(\Delta C_i / C_i)^2 + (\Delta V_i / V_i)^2]$$

where  $\Delta C_i$  is the uncertainty in the concentration  $C_i$  and  $\Delta V_i$  is the uncertainty in the volume  $V_i$ .

So that

$$\text{Uncertainty in Mass Balance} = (T_1 + T_2 + T_3 \dots)^{1/2}$$

**7.0**      **QUALITY ASSURANCE PROVISIONS**

**7.1**      **Responsibility of Inspection**

The chemist in charge of the experiments shall inspect data for reasonableness and completeness.

The technician performing extractions or smears shall inspect records for completeness and recorded data for accuracy.

**7.2**      **Acceptance Criteria**

Experimental data, when accumulated and reviewed shall be acceptable if explanations of experimental phenomena are reasonable and data follow expected behavior or similar chemical and physical systems.

**7.3**      **Material Monitoring**

None

**7.4**      **Equipment Monitoring**

None

**7.5**      **Certification**

This procedure is certified by the review and approval process.

**7.6**      **Quality Control Sample Requirements**

None

**8.0**      **SAFETY**

See document HGD-0004, "Safety and Emergency Plans" for detailed safety requirements for this project.

**9.0**      **NOTES**

None

**10.0**     **ATTACHMENTS AND APPENDICES**

None

**END OF PROCEDURE**



**APPENDIX E3**

**PROCEDURE HGD-0003**

**"PLANNING SAMPLING ACTIVITIES AND SAMPLING"**

## **PLANNING SAMPLING ACTIVITIES AND SAMPLING**

### **1.0 PURPOSE**

This document describes sampling activities for smears and munitions extracts.

### **2.0 SCOPE**

This document applies to field experiments in the Hot Gas Decontamination Project utilizing TVA personnel at Hawthorne, Nevada.

### **3.0 SUMMARY**

Planning activities are described which document which samples to be taken and what solvents will be used.

Munitions are sampled for spiked or residual explosives by solvent rinse or by smear with filter paper dampened with suitable solvent.

### **4.0 REFERENCES**

None

### **5.0 RESPONSIBILITIES**

It is the responsibility of the shift engineer to design the experiment, designate the number of samples to be taken, designate the sampling points, and to document experimental design.

It is the responsibility of the technician to follow this procedure and to adhere to all safety requirements.

### **6.0 PROCEDURE/REQUIREMENTS**

#### **6.1 Prerequisites**

Procedure HGD-0002 "Extraction Experiments" will determine the method for extraction.

**6.2**        **Limitations and Actions**

**6.2.1**       For extraction, the following solvents will be employed:

Acetonitrile - Comp B, TNT, RDX, and HBX

Water - Yellow-D

50/50 mixture of Acetonitrile and Hexane - Comp A

**6.2.2**       Unless otherwise stated in the daily test plan designs, utilize the following quantities of solvent for extractions:

175 millimeter munitions - 500 milliliters of solvent

5 inch and 105 millimeter - 100 milliliters of solvent

3 inch - 50 milliliters of solvent

**6.3.3**       Unless otherwise stated in the daily test plan designs, utilize the following quantities of spike

175 millimeter munitions - 20 g target compound dissolved in 100 ml solvent  
(use larger volumes of solvent if the compound will not freely dissolve)

5 inch and 105 millimeter - 7 g target compound in 50 ml solvent

3 inch - 50 milliliters of solvent - 2.2 g target compound in 25 ml solvent

**6.3**        **Requirements**

**6.3.1**       **Apparatus/Equipment**

Smears - Cotton cloth - sterile bandage quality.

Analytical Balance - Calibrated laboratory balance capable of weighing to 0.0001 g. Calibration traceable to National Institute of Standards and Technology.

Volumetric flasks - various sizes - laboratory grade.

Pipette - Type A volumetric - various sizes

Plug - a machined threaded plug designed to fit each type munition.

Seals - adhesive paper seals used to seal sample bottles after sampling.

Custody Seal - A tamper-proof seal designed for chain of custody evidence.

Temperature Blank - a bottle filled with ethylene glycol or antifreeze. Just before shipping, the temperature of this bottle is measured. Upon receipt, the temperature of this bottle will be measured. In this way, a thermometer need not be inserted in a sample.

#### 6.3.2 Reagents and Standards

Water - laboratory grade deionized water

Acetonitrile - HPLC grade

Hexane - HPLC grade

50/50 mixture of Acetonitrile and Hexane - mix equal volumes of HPLC grade acetonitrile and hexane.

Target compound - an explosive compound supplied at Hawthorne

50 ppm standard. Weigh approximately 50 mg of target compound into a tared 1-liter volumetric flask. More than one compound may be utilized. Bring to the mark with a suitable solvent such as water or acetonitrile. Other concentrations may be prepared in like manner if experimental design calls for them.

5 ppm standard. - Dilute 25 ml of the 50 ppm standard to 250 ml in a volumetric flask. Other concentrations or volumes may be made in like manner if experimental design calls for them.

Ethylene glycol - commercial antifreeze or reagent grade ethylene glycol used as a temperature blank.

**6.4        Calibration**

None

**6.5        Procedure Instructions**

**6.5.1      Devising and Documenting a Sampling Plan**

For each facility load, select one spiked surface at random to be extracted or smeared before the run. ( The purpose of this sample is to verify spiking before the run). Note the location of the other spiked surfaces or munitions. Ensure they are marked so they can be relocated. Note their location on the Sampling Workplan. Document the date, time, munition type, constraints of the run, and other pertinent information on the Workplan. Attach any pertinent drawings for reference. Note any special sampling constraints such as duplicate samples, multiple smears in the same spot, special techniques, whether multiple smears should be separated or placed in the same bottle, etc.

**6.5.2      Spiking**

Dissolve the correct quantity of target compound in the volume of solvent listed in sections 6.2.1 and 6.2.3 above. Use only 100 ml, 50ml, or 25 ml solvent at a time for those situations in which the solvent does not dissolve in 100, 50, or 25 ml solvent. Using a pipette, allow the solution to trickle into the munition to be spiked in such a manner as to cover the largest surface area possible. Allow the solvent to air evaporate between deposits. It may take more than a day for the solvent to evaporate. Rinse the flask and pipette with small quantities of solvent to ensure residue is transferred to the munition. Record all quantities and actions in a field logbook.

**6.5.2      Extract Sampling**

Complete a Field Sampling Sheet utilizing the information on the Sampling Workplan

Place a suitable quantity of solvent into the munition to be extracted. Stopper the munition with a plug.

Rotate and agitate the munition for the time prescribed on the field sheet.

Label the sample container.

Remove the plug and rotate the munition so that the solvent will drain. Collect the solvent in the sample container. Catch as much as possible and allow the surface to drain completely.

Log times, solvents, quantities used, and an estimate of the quantity recovered on the field sheet.

Seal the bottle.

Repeat for the number of times prescribed in the field sheet.

**6.5.3      Making Standard Solutions**

The 50 ppm standard solution and the 5 ppm standard solution shall be made in accordance with the mixing instructions above. All weights and volumes shall be recorded on a Solution Logsheet

**6.5.4      Addition of Quality Control Samples to Sampling Lot**

Once an extraction sampling lot is assembled, add a blank, a 50 ppm standard, and a 5 ppm standard solution for each group of 20 samples or subset thereof. Utilize volumes of the blank and standards similar to the volume of the lot. Utilize the same type bottles, labels, seal, and pen as for the field samples. Also include a labeled bottle with a portion of the spiking solution for verification.

For a smear sampling lot, add a blank smear and two smears to which a measured quantity of 50 ppm standard has been added for each 20 samples or subset thereof. Seal these samples in labeled bottles just as would be done for routine samples. Record the quantity of 50 ppm standard which was used on the Solution Logsheet.

**6.5.5      Random Numbering of Samples**

Samples should be assigned numbers on a random basis to prevent the laboratory from identifying the quality control samples. However, the 10-ml spiking solution sample should not be numbered in this fashion but should be labeled for what it is. Utilize a random number table to provide sample numbers or any other suitable means of numbering the samples so that the laboratory cannot tell which sample is which. For each run, enter the Year, month, date, and two-digit code determined from sequential pairs of number on the random number table. (Any other numbering system which meets the needs of the field team may be utilized.

For example, if a code is needed to indicate what compound is being investigated, it may be added.) Example: For a run on June 14, 1994, the random number table is consulted and a beginning number 22 is chosen. The first sample would be 94-06-14-22. Continue down the random number table taking each sequential pair of digits. Take care to skip duplicate number pairs so that each sample has a unique number.

**6.5.6**      **Chain of Custody Papers and Shipment**

Arrange the samples in numerical order by the random sample number assigned in the step above. Complete the "Chain of Custody" form. Double-check the form for accuracy. Sign and date the "Relinquished by" portion of the form. Pack the samples in plastic bags and then in a cooler filled with ice. Add a temperature blank. Note the temperature on the custody form. Seal the cooler with a custody seal. Attach a copy of the chain of custody papers to the outside of the cooler. Fax a copy to the receiving laboratory. Contact the shipping company and arrange pickup.

**6.5.1**      **Smear Sampling**

(Note: in the following section , it may be helpful to complete as much of the field sheets and labels as is known beforehand.)

Complete a Field Sampling Sheet utilizing the information on the Sampling Workplan

Label a sample bottle with an identifying number or code from the worksheet.

Wear gloves. Change them if contamination of the gloves is suspected or if solvent breakthrough is expected.

Moisten a smear with a suitable solvent. Holding the smear in tongs, vigorously scour the scribed area with the moist smear. Immediately place it into the sample bottle. Rinse the tongs to prevent contamination of the next sample. If the sampling plan requires it, repeat with another smear and place it in the same bottle.

Log all actions, locations, solvent, and number of smears on the Sampling Field Sheet.

Seal the bottle.

**6.6**      **Calculating and Reporting Data**

None

**7.0**      **QUALITY ASSURANCE PROVISIONS**

**7.1**      **Responsibility of Inspection**

It is the responsibility of the engineer to ensure sampling plans are complete and reasonable.

It is the responsibility of the technician to ensure sample documentation is complete, random numbers are assigned without duplication, and that the chain of custody form is correctly completed.

It is the responsibility of the technician to ensure sample documentation is complete, random numbers are assigned without duplication, samples are taken in accordance with the sampling plan, and that the chain of custody form is correctly completed.

**7.2**      **Acceptance Criteria**

The chain of custody form must be completely filled out. All blanks must be completed or a line and "NA" drawn through them. All samples listed on the form must be sealed in the cooler with no extra samples and no omissions.

The cooler must be correctly sealed with evidence tape with no breaks or tears.

**7.3**      **Material Monitoring**

None

**7.4**      **Equipment Monitoring**

None

**7.5**      **Certification**

This procedure is certified by the review and approval process.

**7.6**      **Quality Control Sample Requirements**

None



**8.0**      **SAFETY**

**8.1**      Solvents utilized in this procedure have some hazards associated with them as summarized below. Also reference any Material Safety Data Sheets present in the work area.

**8.1.2**      Acetonitrile is a flammable liquid which is poisonous by ingestion and intraperitoneal routes. It is a skin and severe eye irritant. It is a dangerous fire hazard when exposed to heat, flame, or oxidizers. It reacts with water. OSHA Permissible Exposure Limit is 40 ppm (70 mg/m<sup>3</sup>). The Short Term Exposure Limit is 60 ppm.

**8.1.3**      Methanol is a flammable liquid which is poisonous by ingestion. Some experimental data show that it is poisonous by skin contact. It is mildly toxic by inhalation. Both by ingestion and inhalation it may attack the optic nerve. It is an eye and skin irritant. Death from ingestion of 30 ml has been reported. It is a dangerous fire hazard when exposed to heat, flame, or oxidizers.

**8.1.4**      The solvents should be utilized in a laboratory hood for small objects and in a well ventilated area for large objects. A general rule of thumb is that if an odor is detected, the TLV has been violated. If an odor is detected, wear a respirator.

**8.1.5**      In case of skin contact, wash immediately with soap and water.

**8.1.6**      In case of eye contact, flush immediately with water, holding the eyes open to ensure they are rinsed.

**8.2**      Gloves impenetrable to the particular solvent must be worn when handling it.

**8.3**      The quantities of explosives utilized in this procedure are large enough quantities to constitute a hazard. No flames, sparks, or sparking materials shall be allowed in their presence. When working with the dry powders, electrical grounding shall be utilized to prevent sparks from static electricity. Avoid excess friction or impact.

**9.0**      **NOTES**

**10.0**      **ATTACHMENTS AND APPENDICES**

**END OF PROCEDURE**

Field Sampling Sheet  
Solvent Extractions

Sheet No: \_\_\_\_\_

Solvent: \_\_\_\_\_ Volume: \_\_\_\_\_

Date YY-MM-DD	Random Number xxx	Sample Description
		Spiking Solution - 10 ml
		Blank
		High QC                      Reference
		Low QC                      Reference
		1
		2
		3
		4
		5
		6
		7
		8
		9
		10
		11
		12
		13
		14
		15
		16
		17
		18
		19
		20
		Blank
		High QC                      Reference
		Low QC                      Reference
		21
		22
		23
		24
		25
		26
		27
		28
		29
		30
		31
		32
		33
		34
		35
		36
		37
		38
		39
		40

Reference Planning Sheet Number \_\_\_\_\_  
Notes

Signature: \_\_\_\_\_

Sampling Workplan

Sheet Number \_\_\_\_\_

Scheduled Date: \_\_\_\_\_

Description of Experiment \_\_\_\_\_ Solvent: \_\_\_\_\_

Total number of samples \_\_\_\_\_

Sampling Locations

1	11	21	31
2	12	22	32
3	13	23	33
4	14	24	34
5	15	25	35
6	16	26	36
7	17	27	37
8	18	28	38
9	19	29	39
10	20	30	40

Additional Information

Signature: \_\_\_\_\_

Sheet Number: \_\_\_\_\_

Compound	Mg	Total Volume	Solvent	Name	Date	ID Number	Conc.
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[illegible]

ID Number	MI	Total Volume	Solvent	Name	Date	ID Number	Conc.
Stock	Used					Dilution	

[illegible]

ID Number	MI Used	Sample ID	Name	Date	Total Mg
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[illegible]

CHAIN OF CUSTODY RECORD  
RCRA FACILITY INVESTIGATION

[illegible]

A TABLE OF 14,000 RANDOM UNITS

Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
1	10480	15011	01536	02011	81647	91646	69179	14194	62590	36207	20969	99570	91291	90700
2	22368	46573	25595	85393	30995	89198	27982	53402	93965	34095	52666	19174	39615	99505
3	24130	48360	22527	97265	76393	64809	15179	24830	49340	32081	30680	19655	63348	58629
4	42167	93093	06243	61680	07856	16376	39440	53537	71341	57004	00849	74917	97758	16379
5	37570	39975	81837	16656	06121	91782	60468	81305	49684	60672	14110	06927	01263	54613
6	77921	06907	11008	42751	27756	53498	18602	70659	90655	15053	21916	81825	44394	42880
7	99562	72905	56420	69994	98872	31016	71194	18738	44013	48840	63213	21069	10634	12952
8	96301	91977	05463	07972	18876	20922	94595	56869	69014	60045	18425	84903	42508	32307
9	89579	14342	63661	10281	17453	18103	57740	84378	25331	12566	58678	44947	05585	56941
10	85475	36857	43342	53988	53060	59533	38867	62300	08158	17983	16439	11458	18593	64952
11	28918	69578	88231	33276	70997	79936	56865	05859	90106	31595	01547	85590	91610	78188
12	63553	40961	48235	03427	49626	69445	18663	72695	52180	20847	12234	90511	33703	90322
13	09429	93969	52636	92737	88974	33488	36320	17617	30015	08272	84115	27156	30613	74952
14	10365	61129	87529	85689	48237	52267	67689	93394	01511	26358	85104	20285	29975	89868
15	07119	97336	71048	08178	77233	13916	47564	81056	97735	85977	29372	74461	28551	90707
16	51085	12765	51821	51259	77452	16308	60756	92144	49442	53900	70960	63990	75601	40719
17	02368	21382	52404	60268	89368	19885	55322	44819	01188	65255	64835	44919	05944	55157
18	01011	54092	33362	94904	31273	04146	18594	29852	71585	85030	51132	01915	92747	64951
19	52162	53916	46369	58586	23216	14513	83149	98736	23495	64350	94738	17752	35156	35749
20	07056	97628	33787	09998	42698	06691	76988	13602	51851	46104	88916	19509	25625	58104
21	48663	91245	85828	14346	09172	30168	90229	04734	59193	22178	30421	61666	99904	32812
22	54164	58492	22421	74103	47070	25306	76468	26384	58151	06646	21524	15227	96909	44592
23	32639	32363	05597	24200	13363	38005	94342	28728	35806	06912	17012	64161	18296	22851
24	29334	27001	87637	87308	58731	00256	45834	15398	46557	41135	10367	07684	36188	18510
25	02488	33062	28834	07351	19731	92420	60952	61280	50001	67658	32586	86679	50720	94953
26	81525	72295	04839	96423	24878	82651	66566	14778	76797	14780	13300	87074	79666	95725
27	29676	20591	68086	26432	46901	20849	89768	81536	86645	12659	92259	57102	80428	25280
28	00742	57392	39064	66432	84673	40027	32832	61362	98947	96067	64760	64584	96096	98253
29	05366	04213	25669	26422	44407	44048	37937	63904	45766	66134	75470	66520	34693	90449
30	91921	26418	64117	94305	26766	25940	39972	22209	71500	64568	91402	42416	07844	69618
31	00582	04711	87917	77341	42206	35126	74087	99547	81817	42607	43808	76655	62028	76630
32	00725	69884	62797	56170	86324	88072	76222	36086	84637	93161	76038	65855	77919	88006
33	69011	65797	95876	55293	18988	27354	26575	08625	40801	59920	29841	80150	12777	48501
34	25976	57948	29888	88604	67917	48708	18912	82271	65424	69774	33611	54262	85963	03547
35	09763	83473	73577	12908	30883	18317	28290	35797	05998	41688	34952	37888	38917	88050
36	91567	42595	27958	30134	04024	86385	29880	99730	55536	84855	29080	09250	79656	73211
37	17955	56349	90999	49127	20044	59931	06115	20542	18059	02008	73708	83517	36103	42791
38	46503	18584	18845	49618	02304	51038	20655	58727	28168	15475	56942	53389	20562	87338
39	92157	89634	94824	78171	84610	82834	09922	25417	44137	48413	25555	21246	35509	20468
40	14577	62765	35605	81263	39667	47358	56873	56307	61607	49518	89656	20103	77490	18062
41	98427	07523	33362	64270	01638	92477	66969	98420	04880	45585	46565	04102	46880	45709
42	34914	63976	88720	82765	34476	17032	87589	40836	32427	70002	70663	88863	77775	69348
43	70060	28277	39475	46473	23219	53416	94970	25832	69975	94884	19661	72828	00102	66794
44	53976	54914	06990	67245	68350	82948	11398	42878	80287	88267	47363	46634	06541	97809
45	76072	29515	40980	07391	58745	25774	22987	80059	39911	96189	41151	14222	60697	59583
46	90725	52210	83974	29992	65831	38857	50490	83765	55657	14361	31720	57375	56228	41546
47	64364	67412	33339	31926	14883	24413	59744	92351	97473	89286	35931	04110	23726	51900
48	08962	00358	31662	25388	61642	34072	81249	35648	56891	69352	48373	45578	78547	81788
49	95012	68379	93526	70765	10593	04542	76463	54323	02349	17247	29865	14777	62730	92277
50	15664	10493	20492	38391	91132	21999	59516	81652	27195	48223	46751	22923	32261	85653

**APPENDIX E4**

**PROCEDURE HGD-0004**

**"SAFETY AND EMERGENCY PLANS"**

**1.0** **PURPOSE**

This document describes safety requirements and emergency plans for the Hot Gas Decontamination Project at TVA's Environmental Research Center. See Note 7.1.

This document is not intended to restate all existing safety requirements in place for TVA laboratories, but is intended to describe those additional requirements specific to this project.

**2.0** **SCOPE**

This document applies to work performed at TVA's Environmental Research Center in support of the Hot Gas Decontamination Project. This work will involve experiments and measurements with small quantities of high explosives.

**3.0** **SUMMARY**

In addition to existing TVA safety requirements and existing emergency plans, additional detailed requirements are specified.

**4.0** **REFERENCES**

**4.1** "Manual of Safe Work Practices," October 1990, Resource Development, Tennessee Valley Authority

**4.2** "Chemical Hygiene Plan", National Fertilizer and Environmental Research Center, Revision 1, Issued November 26, 1991

**4.2.1** Appendix I to the "Chemical Hygiene Plan", "Emergency Plan"

**4.3** "Quality Assurance Plan," QA-PLAN, Revision R1, Chemical and Environmental Analysis Section, Tennessee Valley Authority, February 18 10, 1993.

**4.4** "Control of Reagents and Standards," GLP-0006, Revisions R0, General Analytical Laboratory, Tennessee Valley Authority, September 28, 1989.

**5.0** **SAFETY REQUIREMENTS**

The general laboratory safety requirements of references 4.1 and 4.2 apply with the following additions:



**5.1** Defined Work Areas - Work with explosive target compounds for the Hot Gas Decontamination Project shall be limited to the following locations:

- Weighing and desiccating target compounds and mixing standard solutions in room L171 -172.
- Storing and weighing larger quantities of target compounds in room T5.
- Making dilutions and performing initial sample preparation in room L171-172.
- Performing liquid chromatography concentration measurements on samples stored in an autosampler tray in room L173 - 176.
- Performing spiking, smearing, and extraction experiments in room L171 - 172.
- Volatilization of test amounts of target compounds in a tube furnace and collecting them in a sampling train in room L170.

**5.2** Occupancy of Work Areas -

**5.2.1** An analytical area is defined as a ten foot radius around a work area within the same room. Occupancy of analytical areas shall be limited to the following:

- Weighing of standards - one operator and one backup.
- Mixing of standards - one operator and one backup.
- Dilution of solutions, loading of sample vials, sample preparation - one operator and no more than one other person in room L171 - 172 and no more than three other persons in room L172 - 176.

**5.2.2** Occupancy of the tube furnace area shall be one person while the furnace is loaded or unloaded. Ordinarily, the room should be evacuated, locked, and placarded when the experiment is running.

**5.3** General Safety

**5.3.1** As required by the QA Plan (ref. 4.3), all work carried out under this project shall be performed in accordance with written, approved procedures.

- 5.3.2 Gloves, safety glasses and lab coats shall be worn at all times while working with solutions. In addition, face shields shall be worn when working with dry compounds with quantities in excess of one gram.
- 5.3.3 Aisles, doorways, and hallways shall be kept clear to allow for instant exit.
- 5.3.4 Avoid mechanical shock, flame, sparks, static electricity when working with dry compounds. Work on grounding mats when working with dry compounds. Avoid flame and sparks when working with solutions.
- 5.3.5 Dry compounds and solutions shall be stored as close to the floor as possible in their respective storage locations.
- 5.3.6 Solutions and compounds shall be stored and transported in secondary containment ( for example: plastic trays or pans) to limit spills.
- 5.3.7 Inspect storage container threads and frits for dried target compound. Clean threads and frits to prevent any buildup which might detonate due to friction in opening or closing the containers.
- 5.3.8 When weighing dry target compounds do not use of plastic spatulas and weighing dishes. Use metal spatulas and metal weighing dishes instead.
- 5.3.9 Refrigerators used in storing solutions or dry target compound shall be rated as explosion proof. No other materials shall be stored in the refrigerator. The refrigerator shall be locked when not being accessed.
- 5.3.10 Each experiment or procedure step requiring the use of dry target compound should be designed to use the smallest quantity of material practicable.
- 5.3.11 Rooms containing target compounds shall be placarded. They shall be locked after hours to prevent accidental contact with the compounds.
- 5.3.12 Placards for rooms shall state "Explosives - Unauthorized Entry Strictly Prohibited." Placards for equipment such as refrigerators shall state "Explosives - Unauthorized Access Strictly Prohibited." Placards for rooms shall have names and phone numbers for emergency contacts which include the Manager and Materials Custodians.

#### **5.4 Storage of Materials**

**5.4.1** Dry compounds shall be partitioned into approximately one gram quantities and stored in separate containers, each appropriately labeled. These containers shall be stored in spacers or racks which place them at least one inch apart.

**5.4.2** Dry standard materials shall be stored in an explosion-proof container in the storage room (T5). The room shall be kept locked with two persons having key custody, the materials custodians. All access to the room shall be logged. (See form 8.1) A copy of the key shall be maintained with TVA Public Safety with appropriate security controls. All access to the room shall be made by a materials custodian accompanied by a backup who does not enter the room but remains outside to call for help should an incident occur.

- Exception: Small quantities of standard material which are required to be stored in a refrigerator shall be stored in the explosion-proof refrigerator in L171-172.
- Exception: Small quantities of standard material which are required to be stored in a desiccator shall be stored in L171-172.

**5.4.3** Only the quantity of material to be used should be removed from the storage room. It should be weighed into a container and the original container replaced in the storage container.

- Exception: If in certain experiments, entire one-gram containers of dry standard materials need to be removed from T5, they shall be removed for only the length of time required to utilize them in making standards or performing experiments. They shall be kept under lock and key while not being directly utilized in the laboratory and shall be returned to T5 as soon as practicable.

**5.5** Storage of Solutions - Solutions made from standard material shall be stored in an explosion-proof refrigerator in room L171 - 172. The room shall be locked at night and during the day when unattended to prevent accidental contact with the solutions. The refrigerator shall be placarded to warn of the presence of explosive compounds. Solutions with total concentration of target compounds greater than 1% (10,000 ppm) shall be stored in the refrigerated sample storage room T11.

**5.6** Labeling Solutions - Solutions shall be labeled with the compounds present, concentration, and solvent in accordance with the requirements of the QA Plan (ref. 4.3) and GLP-0006 (ref. 4.4). Dry compound containers shall be labeled in accordance with the same requirements.

**5.7 Tube Furnace Experiments**

**5.7.1** While performing tube furnace experiments, doors shall be placarded to prevent accidental access. The door shall not be blocked, however.

**5.7.2** Blast shields shall be used in tube furnace experiments.

**5.7.3** Quantities of target compounds shall be limited to 200 milligrams in tube furnace experiments. Smaller quantities should be used when practical.

**5.7.4** Tube furnace experiments shall be temperature controlled to ensure a long heating time with continued gas flow to drive volatilized compound into the sampling train. At no time should rapid heat be applied which might result in a detonation.

**5.7.5** Following tube furnace runs, joints and cool spots shall be visually inspected for condensation of target compound. After experimental runs or as needed, the interior surface of the exposed components shall be triple-rinsed with a suitable solvent to ensure contamination or residues are eliminated.

**5.8 Sample Storage and Transport**

**5.8.1** Samples shall be stored in a refrigerated storage room located in either T11 or T5. If after analysis solutions are found to contain more than one percent of target compound, they shall be moved to T11 pending shipment back to Hawthorne.

**5.8.2** Samples shall be stored on the floor in plastic trays or pans to prevent dropping and spilling.

**5.8.3** Samples shall be transported between rooms in plastic trays or pans or in rubber buckets.

**5.8.4** Samples shall be returned to the storage area as soon as practical after use.

## **5.9 Liquid Waste**

**5.9.1** Liquid waste should be segregated by target compound insofar as that is possible. Waste containers should be no larger than one liter. Full waste containers should be shipped to Hawthorne for disposal as soon as practical. Pending shipment, waste shall be labeled and stored in the sample storage area in plastic pans or trays on the floor. When waste concentration of target compounds is known to be greater than 1%, it shall be stored in the sample storage area T11.

**5.9.2** Waste containers may be stored in the laboratory while operations are in progress, but should be properly labeled.

**5.9.3** Waste containers should not be left in the laboratory overnight with the exception of the liquid chromatograph discharge stream during automated runs. This discharge container shall be kept in a plastic tray or pan to catch overflows or spills.

**5.9.4** Labels of waste containers shall contain the words "Explosives Process Waste," the name of the solvent or solvents, and the compounds expected to be present.

## **5.10 Solid Waste**

**5.10.1** Solid waste known to be contaminated shall be stored in plastic bags with labels in either T11 or T5 pending shipment to Hawthorne for disposal.

**5.10.2** Solid waste suspected to be contaminated may be accumulated in the laboratory work area in plastic bags. When bags are full, they shall be stored as in 5.10.1

## **5.11 Definition of Clean Glassware**

Glassware may be considered clean after draining, triple rinsing with a suitable organic solvent, and then washing with soap and water. Collect the organic rinses and treat them as liquid waste as in 5.9.

## **5.12 Chemical Hygiene Committee Review**

This Safety and Emergency Plan and the work under its scope shall receive a hazard review by the Chemical Hygiene Committee of Analytical Support Services as specified in the Chemical Hygiene Plan (ref. 4.2) . Changes in quantities utilized in experiments shall be reviewed as specified by the Chemical Hygiene Plan.

A review by the Chemical Hygiene Officer for the Center, the Chemical Hygiene Officer for the Laboratory, and the Manager for the Laboratory may be substituted for the hazard review by the Chemical Hygiene Committee during times of organizational transition.

**6.0      EMERGENCY PLAN**

The requirements of reference 4.2.1 apply with the following additions specific to this project.

**6.1      Cooling loss**

No cooling lines are proposed in the experimental setup.

**6.2      Vacuum loss**

If vacuum is used to move the gas stream through the tube furnace, vacuum failure should be followed by immediate shutdown of the furnace power supply.

**6.3      Power loss**

If power loss occurs due to line failure, disconnect or shut down equipment so that it does not come back on unexpectedly.

Power loss in tube furnace experiments is not expected to provide additional hazards although deposition of target compound can occur on interior surfaces. In this case, the furnace should be disassembled and cleaned with a suitable solvent.

Power loss in liquid chromatographs could result in deposit or condensation of target compounds in interior sample pathways. The unit should be cleaned by injecting solvent until a good baseline is observed unless chromatogram traces indicate no appreciable amount of target compound was present in the machine at the time of power failure.

**6.4      Spills**

**6.4.1      Liquid Spills of Organic Solvents Such as Acetone, Ethanol, Methanol, and Acetonitrile**

**6.4.1.1      Spills should be contained in the secondary containment described throughout section 5 above.**

**6.4.1.2** Small spills that miss containment should be cleaned up by paper towels. Place contaminated towels in a plastic bag in a hood while cleanup work is in progress.

**6.4.1.3** Spills larger than 500 ml should be cleaned up with an organic spill kit. The kits are located on the carts near the stairwell.

**6.4.1.4** Dispose of paper towels and solid waste as described in section 5.10.

**6.4.1.5** For spills larger than 4 liters which miss containment, breathing apparatus may be required to enter the area. Only properly trained, medically certified and tested personnel may respond. If a spill kit cannot be put into use within five minutes spark and fire hazards then become the controlling danger. Do not turn off or turn on equipment. Evacuate the laboratory and contact the hazard response team for the building. Notify neighboring rooms and the floors above and below the spill area. Evacuate these rooms if the spill cannot be contained within 30 minutes or if a noticeable odor is present in them.

**6.4.2 Solid spills**

Since quantities of target compound are limited to 1 gram or less, clean spills of solid material by sweeping gently with a paper towel into a plastic bag. Follow this by cleaning the surface with soap and water. Dispose of solid waste as in section 5. For larger spills, contact the hazard response team for the building.

**6.5 Hood failure**

Halt any reaction or operation occurring in the hood. Cap containers. Leave the work area and notify the supervisor and Chemical Hygiene Officer. Lock the room to prevent unauthorized entrance. Clear the hood of hazards before repair personnel begin work where they might come in contact with them. After repair, ensure the hood is operational before starting operations in it.

**6.6 Fire**

Leave the work area. Notify Public Safety. Pull the local fire alarm. Notify the supervisor.

**6.7 Explosion**

Leave the work area. Notify Public Safety. Pull the local fire alarm. Notify the supervisor.

**6.8 Accidental Personal Exposure**

Wash the exposed area immediately with soap and warm water. If eyes are exposed, call loudly for help and use the nearest eye wash. If large areas are exposed, utilize the nearest safety shower. Follow this by a shower with soap and warm water. If clothing is contaminated, remove it immediately and do not replace it until after it is laundered.

**6.9 Runaway Reactions**

Leave the work area. Notify Public Safety. Notify the supervisor.

**6.10 Emergency Shutdown Procedures**

To shut down the tube furnace assembly, disconnect the power.

To shut down the liquid chromatograph, turn off the uninterruptible power supply connected to the unit.

**6.11 Unrelated Building Emergencies**

If in the course of analysis or experiments an unrelated building emergency occurs such as a fire drill, tornado warning or the like which requires evacuation of the workspace

- Disconnect power for any activity which would cause a hazard if power failed and came back on. (Example: Tube furnace experiment)
- Evacuate the area as instructed.

**6.11 Notifications**

Following any incidents listed above, the supervisor should be notified. Do not delay in obtaining medical help or emergency response in order to find the supervisor first. Likewise, notify the Chemical Hygiene Officer and the Manager of Support Services.

**7.0 NOTES**

**7.1** The compounds utilized in this project in the quantities proposed and under routine laboratory handling conditions may be safely weighed, dissolved, and analyzed. The intent of this document is to provide an additional margin of safety to ensure there is no explosion or fire. Quantities are limited to provide even greater margin of safety so that if there were an explosion, its effects would be minimized.

END OF DOCUMENT



**APPENDIX E5**

**PROCEDURE HGD-0005**

**"USE OF EXPLOSIVES STORAGE ROOM"**

**1.0** **PURPOSE**

This procedure gives the protocols and controls for entry to the explosives storage room (T5) and for use of the compounds stored there.

**2.0** **SCOPE**

This applies to entry to the explosives storage room in T5 and to access to the explosives storage container.

**3.0** **SUMMARY**

Entry to the storage room is logged. Safety controls are specified. Use or removal of the dry solids stored there is logged.

**4.0** **REFERENCES**

**4.1** HGD-0004 "Safety and Emergency Plans", Tennessee Valley Authority, Analytical Laboratory, Support Services, Environmental Research Center, Muscle Shoals, Alabama

**4.2** CP-0001, "Measurement and Test Equipment Control and Calibration," Tennessee Valley Authority, Chemical and Environmental Analysis Section, Revision R2, September 20, 1993.

**5.0** **RESPONSIBILITIES**

**5.1** It is the responsibility of the person performing this procedure to adhere to the safety requirements herein, to document all access to the room, and to document all use of the target compounds stored in the room.

**5.2** It is the responsibility of the Manager, Analytical Laboratory Support Services to ensure this procedure is followed. It is the responsibility of the Manager to appoint materials custodians.

**6.0** **PROCEDURE/REQUIREMENTS**

**6.1** **Prerequisites**

**6.1.1** The manager shall appoint two persons in the organization to be materials custodians. These individuals shall have copies of the keys to the room and to padlock on the storage container. Another key set shall be maintained with Public Safety. The manager shall have a fourth key set.

- 6.1.2 The materials custodian performing this procedure must be thoroughly familiar with the safety requirements of HGD-0004.
- 6.1.3 Public Safety shall be instructed to not allow access to the storage room in case of fire. The storage container is designed to withstand any explosion which might occur.
- 6.1.4 Public Safety shall be instructed to communicate any use of the keys controlled by them to Joe Hoagland.
- 6.1.5 Materials custodians shall have been trained in General Explosives Safety, in the contents of HGD-0004, and in the contents of this Procedure. This training shall be documented.

## **6.2 Limitations and Actions**

- 6.2.1 The explosive storage room and the steel container in it are to be used to store dry target compounds which are not being used directly in experiments. Those dry compounds, usually standard materials, which must be refrigerated may be stored elsewhere as described in HGD-0004. Small quantities (less than one gram) which must be desiccated at all times may be stored elsewhere as described in HGD-0004.
- 6.2.2 All the requirements of HGD-0004 apply to this procedure.
- 6.2.3 In the event of the absence of the materials custodians, the Manager, ALSS may assign another individual who has had the proper training and is thoroughly familiar with HGD-0004 and this procedure to perform the work herein.
- 6.2.4 The emergency backup person shall have been trained in the contents of this procedure before serving in that capacity.
- 6.2.5 This procedure and HGD-0004 are written to allow up to twenty grams of explosive material to be stored in the storage room. If that amount is to be exceeded, this procedure and HGD-0004 must be reviewed and modified as necessary.
- 6.2.6 A lab coat, gloves, and a face shield are required for entry into the storage area.

**6.2.7** Access to the room for other purposes than to open the explosives storage container and to work with the compounds stored in it is permitted if a backup is present. This access need not be by the materials custodian, but must be logged. An example of this access would be the routine monthly balance check.

### **6.3 Requirements**

#### **6.3.1 Apparatus/Equipment**

**6.3.1.1** Explosives storage container - a steel container, painted red, labeled with the word "Explosives". This container must have no source of heat, flame, or sparks. The container shall be padlocked when not being accessed.

**6.3.1.2** Analytical balance - a balance capable of weighing to 0.1 mg with calibration traceable to the National Institute of Standards and Technology.

**6.3.1.3** Logbook - A bound logbook with sequentially numbered pages. The logbook shall have carbon paper or another means of duplicating each page as it is written. Completed pages shall be removed and stored separately. The logbook shall be stored in room T5 until the project is complete. Pages of the logbook shall be designed to list all the bottles of target compound stored in the room and their use. Complete traceability of each use of the compounds and ultimate disposal shall be provided.

**6.3.1.4** Inventory Cards - Should the inventory increase to the size that a logbook cannot be readily used to provide traceability, an inventory card system shall be utilized which provides information on each bottle of compound including source, identification code, use, quantity, dates, and ultimate disposal. References below to "logbook" for inventory control may be understood to apply to inventory cards. Inventory cards may be stored outside the room for safekeeping, but should be carried into the room so that data may be written directly on them rather than on another piece of paper and then transferred.

#### **6.3.2 Reagents and Standards**

**6.3.2.1** Target Compounds - An explosive compound or byproduct to be studied as provided by USAEC.

**6.4 Calibration**  
None

**6.5 Procedure Instructions**

**6.5.1 Key Control**

**6.5.1.1** The materials custodians will keep one copy of the key to the room and the key to the padlock on his or her person. The key will not be kept locked in a desk. Transfer of the keys and custody shall be done by the manager. Any transfer of responsibility shall be logged in the explosives room logbook. The keys shall never be left unattended.

**6.5.1.2** A set of keys shall be kept with Public Safety. The keys shall have a tag attached which clearly states "Explosives Storage Room - Limited Access." Any access to this key shall be logged with public safety.

**6.5.1.3** A set of keys shall be assigned to the Manager. These keys may be kept locked in a secure place or may be kept on his person. The keys shall never be left unattended.

**6.5.2 Addition of New Compounds to the Storage Area**

Partition the newly received compound into approximately one gram quantities in suitable containers for storage. Label each bottle with a unique identifier and log the numbers in the logbook. Place the bottles in a rack with approximately one inch space between each bottle. Place the rack in the explosive storage container.

**6.5.3 Room Access**

**6.5.3.1** As required in HGD-0004, only one person enters T5. This must be a materials custodian. Another person, the emergency backup, shall remain present outside the storage area to call for help in the event of an incident. This second person may be any employee who is suitably trained. The emergency backup stays in the hallway outside the room and away from the door so as to avoid flying glass in event of an accident. See attachment 1.

**6.5.3.2** All entry into the room must be logged in the explosives room logbook. The date, time, person's name, and reason shall be logged.

**6.5.4 Use of Compounds**

**6.5.4.1** Handling of compounds in the storage room should be limited to weighing the compound into a container which will then be removed from the room.

**6.5.4.2** Weigh the compound. Log the weights, identification numbers, proposed use of the compound and other pertinent data.

**6.5.5 Removal of Compounds**

**6.5.5.1** In the case that a storage container for a compound must be removed from the storage room for an experiment, the action and its reason shall be logged. Estimated quantity to be used shall also be logged.

**6.5.5.2** Transport the container singly in a plastic pan or bucket to the location for the experiment.

**6.5.5.3** While utilizing the compound in an experiment, do not leave it unattended. Store the bottle under lock and key if it is to be left in the room for the experiment. Return the bottle to the storage container as soon as possible. Never leave the bottle outside the storage container overnight.

**6.5.5.4** When the bottle is returned to the storage room, log its return. Log the amount actually used and its purpose. If the compound was used up in the experiment, log this in the logbook as well.

**6.6 Calculating and Reporting Data**  
None

**7.0 QUALITY ASSURANCE PROVISIONS**

**7.1 Responsibility of Inspection**  
Is the responsibility of the group leader to inspect logs and records to ensure they are accurate and complete.

It is the responsibility of the Quality Assurance Officer to perform inspections of the records produced in the performance of this procedure.

**7.2 Acceptance Criteria**

All access to the room and actions involving use of the materials shall be logged. Data shall be logged in enough detail to reconstruct any use of the compounds. Inventory records shall completely describe each bottle of compound.

**7.3 Material Monitoring**

None

**7.4 Equipment Monitoring**

Balances are monitored in accordance with procedure CP-0001.

**7.5 Certification**

This procedure is certified by performing a walk-through using an inert compound.

**7.6 Quality Control Sample Requirements**

None

**8.0 SAFETY**

Safety requirements are specified in detail in HGD-0004.

**9.0 NOTES**

None

**10.0 ATTACHMENTS AND APPENDICES**

Attachment 1 - Instructions for Backup

Instructions for Emergency Backup - Access to Storage Room

1. Remain in hallway away from the door while the materials custodian works in T5. The door should be blocked open or unlocked during use.
2. Should an emergency occur, call Public Safety at **8911** or **2444**

The nearest telephone will be in one of the offices in the main hallway.

3. Provide any immediate assistance needed by the person in T5 including help using the safety eye wash. Assist them holding their eyes open and call loudly for help.

4. Other Phone Numbers

Team Leader - Joe Hoagland - 2108

Chemical Hygiene Officer - Bill Rogers - 3774

Materials Custodian - David Phillips - 3358

Materials Custodian - Jon Wilson - 2644

Group Secretary - Donna Walters - 2099

END OF PROCEDURE



**APPENDIX E6**

**PROCEDURE HGD-0006**

**"METHOD DETECTION LIMITS"**

**1.0**        **PURPOSE**

This procedure describes the method for calculating method detection limits.

**2.0**        **SCOPE**

This procedure applies only to work done for the Hot Gas Decontamination Project.

**3.0**        **SUMMARY**

At least seven replicate samples are taken through the entire analytical process. The standard deviation is calculated and multiplied by the appropriate t-factor.

**4.0**        **REFERENCES**

Title 40, Code of Federal Regulations, Part 136, Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit " - Revision 1.1.

**5.0**        **RESPONSIBILITIES**

**5.1**        It is the responsibility of the analyst to perform measurements and calculations in accordance with this procedure, to review results, and to report anomalous results to the supervisor.

**5.2**        It is the responsibility of the supervisor to ensure work is performed in accordance with this procedure and to review results.

**6.0**        **PROCEDURE/REQUIREMENTS**

**6.1**        **Prerequisites**

Equipment shall have been set up and configured in accordance with the appropriate analytical procedure.

**6.2**        **Limitations and Actions**

None

**6.3**        **Requirements**

**6.3.1**     **Apparatus/Equipment**

See the appropriate analytical procedure.

**6.3.2 Reagents and Standards**

See the appropriate analytical procedure.

**6.4 Calibration**

See the appropriate analytical procedure.

**6.5 Procedure Instructions**

Perform the method detection measurements and calculations in accordance with Attachment 10.1

**6.6 Calculating and Reporting Data**

Perform calculations in accordance with Attachment 10.1

**7.0 QUALITY ASSURANCE PROVISIONS**

**7.1 Responsibility of Inspection**

The analyst and supervisor shall inspect data for consistency and reasonableness.

**7.2 Acceptance Criteria**

The concentration of analyte should be between one and five times the method detection limit.

**7.3 Material Monitoring**

None

**7.4 Equipment Monitoring**

None

**7.5 Certification**

This procedure is certified by the review and approval process.

**7.6 Quality Control Sample Requirements**

None

**8.0 SAFETY**

See the appropriate analytical procedure.

**9.0 NOTES**

None

10.0 ATTACHMENTS AND APPENDICES

10.1 - 40 CFR 136 Appendix B.

## 10.0 ATTACHMENTS AND APPENDICES

## 10.1 - 40 CFR 136 Appendix B.

## APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

*Definition*

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

*Scope and Application*

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

*Procedure*

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to

be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each

## 10.1 - Continued

through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance ( $S^2$ ) and standard deviation ( $S$ ) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} \left[ \sum_{i=1}^n X_i^2 - \frac{\left( \sum_{i=1}^n X_i \right)^2}{n} \right] \quad S = (S^2)^{1/2}$$

where:

$X_i$ :  $i=1$  to  $n$ , are the analytical results in the final method reporting units obtained from the  $n$  sample aliquots and  $\Sigma$  refers to the sum of the  $X$  values from  $i=1$  to  $n$ .

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1, 1-\alpha = 0.99)} (S)$$

where:

MDL = the method detection limit

$t_{(n-1, 1-\alpha = 0.99)}$  = the student's  $t$  value appropriate for a 99% confidence level and a standard deviation estimate with  $n-1$  degrees of freedom. See Table.

$S$  = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ( $\chi^2/df$ ).

$$LCL = 0.64 MDL$$

$$UCL = 2.20 MDL$$

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use  $S^2$  from the current MDL calculation and  $S^2$  from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger  $S^2$  into the numerator  $S_A^2$  and the other into the denominator  $S_B^2$ . The com-

puted F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: If  $S_A^2/S_B^2 < 3.05$ , then compute the pooled standard deviation by the following equation:

$$S_{\text{pooled}} = \left[ \frac{6S_A^2 + 6S_B^2}{12} \right]^{1/2}$$

If  $S_A^2/S_B^2 > 3.05$ , respoke at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the  $S_{\text{pooled}}$  as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681 (S_{\text{pooled}})$$

where 2.681 is equal to  $t_{(12, 1-\alpha = 0.99)}$ .

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$LCL = 0.72 MDL$$

$$UCL = 1.65 MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS'  $t$  VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates	Degrees of freedom (n-1)	$t_{(n-1, 0.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
∞	∞	2.328

#### Reporting

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

**END OF PROCEDURE**

## **APPENDIX F**

### **REFERENCES**

## REFERENCES

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